QUALITY ASSURANCE PROJECT PLAN FOR THE ALLIED PAPER PLAINWELL No. 2 DAM PRP PCB REMOVAL SITE PLAINWELL, ALLEGAN COUNTY, MICHIGAN NPL STATUS: NPL

Prepared for

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

by

WESTON SOLUTIONS, INC.

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QAPP Worksheet #1
Title and Approval Page

Site Name/Project Name: Allied Paper Plainwell Impoundment & No. 2 Dam Potentially Responsible Party (PRP) Polychlorinated Biphenyl (PCB) Removal Site
Site Location: Plainwell, Allegan County, Michigan

Document Title: Quality Assurance Project Plan (QAPP) for the Allied Paper Plainwell Impoundment & No. 2 Dam PRP PCB Removal Site

Lead Organization: United States Environmental Protection Agency (U.S. EPA) Region V

Preparer's Name and Organizational Affiliation:

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(2) Lisa Graczyk, WESTON START
(3) Linda Korobka, WESTON START
(4) Chris Lantinga, WESTON START

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Preparation Date (Month/Day/Year): May 2010

(1) Investigative Organization's Project Manager (Signature): Chris Lantinga

(Printed Name, Organization, and Date): Chris Lantinga, WESTON START, May 2010

(2) Investigative Organization's Project QA Officer (Signature): Lisa Graczyk

(Printed Name, Organization, and Date): Lisa Graczyk, WESTON START, May 2010

(3) Lead Organization's Project Manager (Signature): Sam Borries

(Printed Name, Organization, and Date): Sam Borries, U.S. EPA Region V, May 2010

Approval Signatures: See Worksheet #4
Signature

Printed Name/Title/Date

Approval Authority

Other Approval Signatures: See Worksheet #4
Signature

Printed Name/Title/Organization/Date

Document Control Number: 819-2E-AGLH

QAPP Worksheet #2
QAPP Identifying Information

Site Name/Project Name: Allied Paper Plainwell Impoundment & No. 2 Dam PRP PCB Removal Site
Site Location: Plainwell, Allegan County, Michigan
Site Number/Code: MID006007306
Operable Unit: Not Applicable (NA)
Contractor Name: Weston Solutions, Inc.
Contractor Number: EP-S5-06-04
Contract Title: Superfund Technical Assessment and Response Team (START)
Work Assignment Number: S05-0002-0909-022

1. Identify guidance used to prepare QAPP:
   Uniform Federal Policy for Quality Assurance Project Plans

2. Identify regulatory program: U.S. EPA Region V, Emergency Response Branch

3. Identify approval entity: U.S. EPA Region V

4. Indicate whether the QAPP is a **generic** or a project-specific QAPP (circle one)

5. List dates of scoping sessions that were held:
   August 11, 2009 – Mike Ribordy (U.S. EPA), Paul Bucholtz (MDNRE), Chris Lantinga (WESTON START), Jay Rauh (WESTON START), and the representatives of the PRP and its contractors.

6. List dates and titles of QAPP documents written for previous site work, if applicable:

Title	Received Date
QUALITY ASSURANCE PROJECT PLAN FOR THE ALLIED PAPER PRP PCB REMOVAL SITE Rev. 0.0	October 2008

7. List organizational partners (stakeholders) and connection with lead organization:
   STATES: Michigan Department of Natural Resources and Environmental (MDNRE)
   Federal Agencies: U.S. EPA, U.S. Fish and Wildlife Service (U.S. FWS), and the National Oceanic and Atmospheric Administration (NOAA)

8. List data users:
   U.S. EPA Region V On-Scene Coordinator (OSC) and MDNRE

9. If any required QAPP elements and required information are not applicable to the project, then circle the omitted QAPP elements and required information on the attached table. Provide an explanation for their exclusion below:

Circle QAPP elements and required information that is not applicable to the project. Provide an explanation in the QAPP.

Required QAPP Element(s) and Corresponding QAPP Section(s)	Optional QAPP Worksheet # in QAPP Workbook	Required Information
	Project Management and Objectives	
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2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	2 2	- Table of Contents - QAPP Identifying Information
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	3 4	- Distribution List - Project Personnel Sign-Off Sheet
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	5 6 7 8	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table
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2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	11 12	- Site-Specific PQOs - Measurement Performance Criteria Table
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3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	Appendix B 23 24 25	- Analytical SOPs - Analytical SOP References Table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table
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5.3.3 Amounts and Types of Data Appropriate for Streamlining		

QAPP Worksheet #3
Distribution List

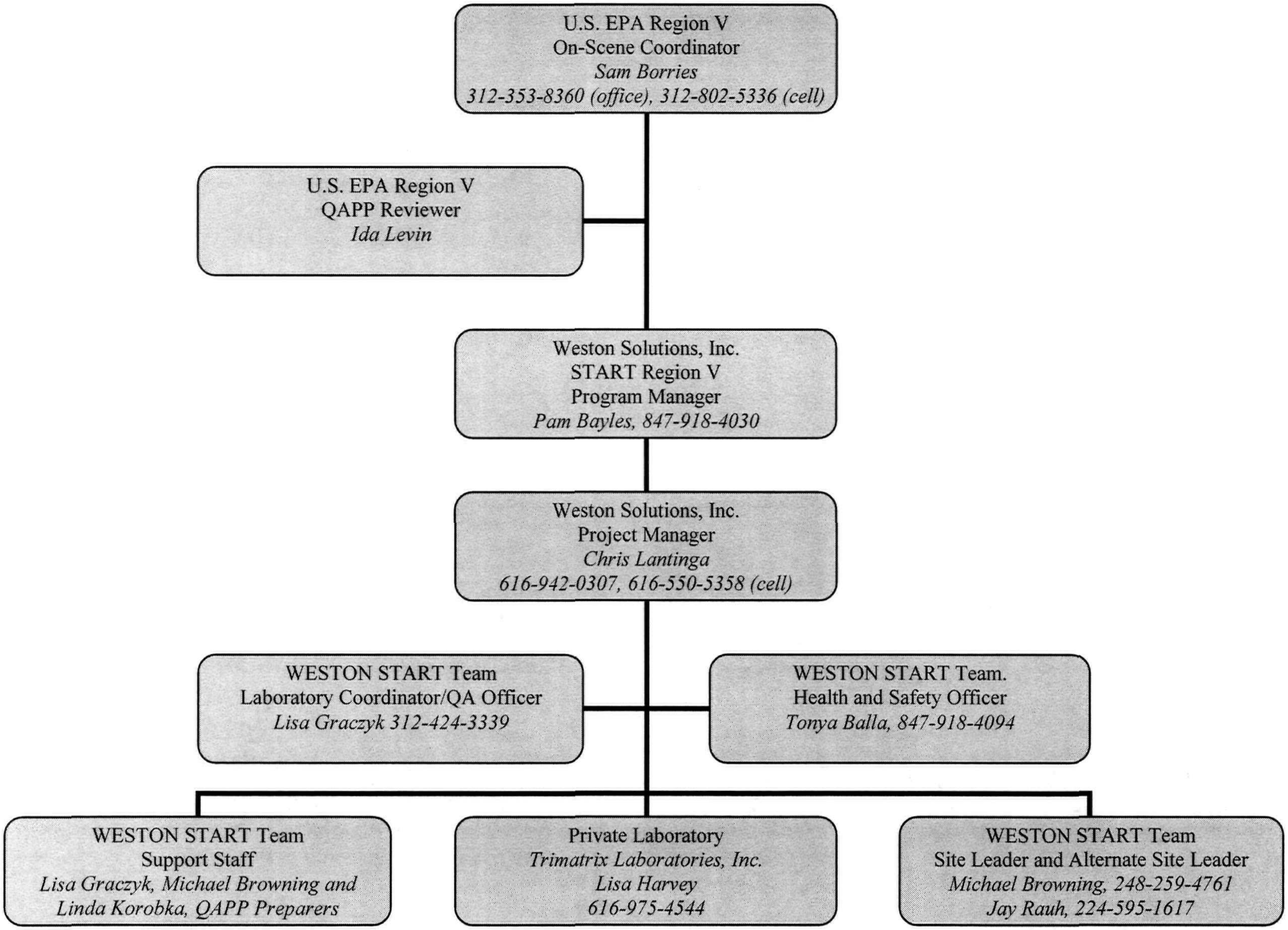
QAPP Recipients	Title	Organization	Telephone Number	Fax Number	E-mail Address	Document Control Number
Sam Borries	OSC	U.S. EPA Region 5	312-353-8360	312-353-9176	borries.samuel@epa.gov	819-2E-AGLH
Paul Bucholtz	MDNRE Representative	MDNRE	517-373-8174	517-335-4887	bucholtp@michigan.gov	819-2E-AGLH
Chris Lantinga	Project Manager	WESTON START	616-942-0307	517-381-5921	christopher.lantinga@westonsolutions.com	819-2E-AGLH
Michael Browning	Site Leader, QAPP Preparer	WESTON START	248-259-4761	313-739-2501	mbrowning@dynamac.com	819-2E-AGLH
Jay Rauh	Alternate Site Leader	WESTON START	312-424-3315	312-424-3330	jay.rauh@WestonSolutions.com	819-2E-AGLH
Linda Korobka	QAPP Preparer/ QA/QC Reviewer	WESTON START	517-381-5936	517-381-5921	linda.korobka@WestonSolutions.com	819-2E-AGLH
Lisa Graczyk	Project QA Officer, Lab Coordinator	WESTON START	312-424-3339	312-424-3330	lgraczyk@dynamac.com	819-2E-AGLH
Ida Levin	Field Services Section (FSS) QAPP Reviewer	U.S. EPA	312-886-6254	312-353-9176	levin.ida@epa.gov	819-2E-AGLH

QAPP Worksheet #4
Project Personnel Sign-Off Sheet

Organization:	Weston Solutions, Inc.

Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Sam Borries	OSC	312-353-8360	[illegible]	5-7-10
Chris Lantinga	Project Manager	616-942-0307	[illegible]	5-1-10
Michael Browning	Site Leader, QAPP Preparer	248-259-4761	Michael J Browning	5/1/10
Jay Rauh	Alternate Site Leader	312-424-3315	[illegible]	5/7/10
Linda Korobka	QAPP Preparer/ QA/QC Reviewer	517-381-5936	Linda Korobka	5-4-10
Lisa Graczyk	Project QA Officer, Lab Coordinator	312-424-3339	Lisa Graczyk	4-19-10
Ida Levin	U.S. EPA FSS QAPP Reviewer	312-886-6254	Ida Levin	6/10/10

QAPP Worksheet #5
Project Organizational Chart



QAPP Worksheet #6
Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, pathways, etc.)
Project scope changes	OSC	Sam Borries	312-353-8360	The OSC will inform the WESTON Project Manager of any project scope changes. The project manager will in turn inform the START program manager of the changes.
Approval of QAPP Amendments	U.S. EPA FSS QAPP Reviewer	Ida Levin	312-886-6254	Approval of all QAPP amendments will be by the FSS QA Reviewer prior to the changes being implemented.
Management of required project tasks	Project Manager	Chris Lantinga	616-942-0307	The WESTON Project Manager will inform the appropriate WESTON project staff (field and non-field) of tasks to complete and the required completion date. The WESTON project staff will communicate with the Project Manager of task progress and resources/information required to complete tasks.
Field corrective actions or delays to field work	Site Leader, Alternate Site Leader	Michael Browning, Jay Rauh	248-259-4761, 224-595-1617	The Site Leader or Alternate Site Leader will inform the Project Manager of any delays or changes to field work, by telephone. The Project Manager will inform the OSC by telephone.
Weekly field updates	Site Leader, Alternate Site Leader	Michael Browning, Jay Rauh	248-259-4761, 224-595-1617	The site leader will inform the Project Manger of weekly field progress, by telephone or e-mail. The Project Manager will then inform the OSC of field work progress, by telephone or email.
Reporting of Laboratory Data Quality Issues	Laboratory Project Chemist	Lisa Harvey, TriMatrix	616-975-4544	The laboratory project manager will inform Lisa Graczyk, the laboratory coordinator, of any issues related to data quality upon receipt of samples or during analyses.

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (Timing, pathways, etc.)
Distribution of analytical data	Laboratory Coordinator	Lisa Graczyk	312-424-3339	The laboratory coordinator will receive all deliverables from the laboratory and distribute them to the Project Manager and data validator. The Project Manager will distribute data and data validation reports to the OSC. The OSC will distribute the data and to other interested parties (MDNRE).
Recommendations to stop work and initiation of corrective actions	QA Officer/OSC/Project Manager	Lisa Graczyk/ Sam Borries/ Chris Lantinga	312-424-3339/ 312-353-8360/ 616-942-0307	The QA Officer, Project Manager, and OSC all have the authority to stop work and initiate corrective actions should there be a reason to do so. Whoever stops the work or initiates corrective actions will inform the Site Leader (or Alternate Site Leader) and Project Manager immediately. The Project Manager will ensure that the QA Officer and OSC are informed of the stop work and corrective actions.

QAPP Worksheet #7
Personnel Responsibilities and Qualifications Table

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Sam Borries	OSC	U.S. EPA Region V	The OSC has overall project authority and directs the project manager regarding the tasks required to meet project objectives.	
Ida Levin	QA/QC Officer	U.S. EPA Region V	The U.S. EPA FSS QAPP reviewer is responsible for reviewing and approving the project-specific QAPP (and any amendments) prior to its implementation.	
Pamela Bayles	START Region V Program Manager	WESTON START Team	The START Program Manager is responsible for ensuring the quality of work performed under the Region V START III contract. The START Program Manager interfaces directly with the U.S. EPA Contracting Officer and Project Officer, and has overall responsibility and direction for task assignments.	M.E.M. (Masters in Environmental Management), Air and Water Resources; B.S., Biology; over 18 years experience
Chris Lantinga	Project Manager	WESTON START Team	The project manager is responsible for managing all aspects of the project, WESTON project personnel, and subcontractors. The project manger interfaces directly with the U.S. EPA OSC regarding all project tasks.	B.A. Engineering/Geology, B.S. Civil Engineering, over 17 years of experience
Michael Browning	Site Leader, QAPP Preparer	WESTON START Team	(1) The site leader manages all work performed in the field. The site leader interfaces directly with the project manager regarding field tasks and any issues that arise while in the field. (2) Prepares the project QAPP.	M.S., Natural Resources Policy, B.S., Environmental Policy, over 10 years experience
Jay Rauh	Alternate Site Leader	WESTON START Team	The alternate site leader manages all work performed in the field when the site leader is not on site. The alternate site leader interfaces directly with the project manager regarding field tasks and any issues that arise while in the field.	B.S., Environmental Geography, over 6 years experience

Name	Title	Organizational Affiliation	Responsibilities	Education and Experience Qualifications
Linda Korobka	QAPP Preparer and QA/QC Reviewer	WESTON START Team	The QAAP preparer writes the project QAPP and reviews QA/QC documents associated with the project.	B.S., Chemistry, over 21 years of environmental experience
Tonya Balla	Health and Safety Officer	WESTON START Team	The health and safety officer approves the Health and Safety Plan and provides guidance to field personnel on health and safety issues.	B.S., Environmental Engineering; over 16 years experience
Lisa Graczyk	Laboratory Coordinator/QA Officer	WESTON START Team	The Laboratory Coordinator is responsible for the procurement of the contracted laboratory and is the main interface with the laboratory regarding project deliverables and QA/QC aspects of the analyses. The QA Officer reviews the project QAPP and has overall responsibility for project QA.	B.S. Chemistry, over 17 years experience

QAPP Worksheet #8
Special Personnel Training Requirements Table

Project Function	Specialized Training By Title or Description of Course	Training Provider	Training Date	Personnel / Groups Receiving Training	Personnel Titles / Organizational Affiliation	Location of Training Records / Certificates[1]
Boating Operations	Michigan Online Boating Safety Course	MDOT	4/22/08	Site Leader (Browning)	WESTON START	Site Files
Field Sampling Activities	40-Hour OSHA HAZWOPER Training and Recurrently Annual 8-hour refreshers	WESTON	Various	Site Leader and Alternate Site Leader	WESTON START	WESTON's web-based EHS Track

[1] If training records and/or certificates are on file elsewhere, document their location in this column. If training records and/or certificates do not exist or are not available, then this should be noted.

QAPP Worksheet #9
Project Scoping Session Participants Sheet

Project Name: Allied Paper Plainwell Impoundment & No. 2 Dam PCB Removal Site Projected Date(s) of Sampling: June2007 – 12/1/2010 (Projected) Project Manager: Chris Lantinga, Weston Solutions, Inc.				Site Name: Allied Paper Plainwell Impoundment & No. 2 Dam PRP PCB Removal Site Site Location: Plainwell, Allegan County, Michigan	
Date of Session: August 11, 2009, May 5, 2010 **Scoping Session Purpose:** To provide an overview of the operations for the Plainwell No. 2 dam project for 2009 and 2010.					
Name	**Title**	**Affiliation**	**Phone #**	**E-mail Address**	**Project Role**
Sam Borries	U.S. EPA OSC	USEPA	(312) 802-5336	borries.samuel@epamail.epa.gov	Project Authority
Paul Bucholtz	Environmental Quality Analyst	MDNRE	(517) 373-8174	bucholtzp@michigan.gov	Project Manager
Chris Lantinga	Project Manager	WESTON	(517) 381-5930	christopher.lantinga@westonsolutions.com	Project Manager
Mike Browning	Site Leader	WESTON	248-259-4761	mbrowning@dynamac.com	Site Leader
Jay Rauh	Alternate Site Leader	WESTON	(312) 424-3315	jay.rauh@westonsolutions.com	Alternate Site Leader

Comments/Decisions:

Action Items:

Consensus Decisions:

QAPP Worksheet #10
Problem Definition

Problem Definition and Background: From the 1950s to the mid-1970s, paper mills in the Plainwell, Michigan area used polychlorinated biphenyls (PCBs) in the production of carbonless copy paper. These paper mills released these PCBs, in their waste water, into the Kalamazoo River and the areas of the river known as the Plainwell Impoundment (which was created from the building of the Plainwell Dam on the Kalamazoo River in the early 1900s) and Plainwell #2 Dam.

Following the Federal government's ban on the production of PCBs in 1976, the use of PCBs in the production of carbonless copy paper ended. However, given the persistent and toxic nature of PCBs in the environment, and their ability to accumulate in the tissues of wildlife, several investigations of the sediment and soil in the Kalamazoo River and the Plainwell Impoundment took place from 1993 to 2008. Given the results of these studies (which are summarized below), the U.S. EPA determined that the concentrations of PCBs in the sediment, river bank soil, historical oxbow channel, and floodplain soil of the former Plainwell Impoundment and Plainwell #2 Dam pose an imminent and substantial danger to both human and ecological receptors. Consequently, U.S. EPA initiated a time-critical removal action (TCRA) to address the contamination existing in the Plainwell Impoundment, and subsequently, the Plainwell #2 Dam along the Kalamazoo River.

The Plainwell No. 2 Dam Area is located on the Kalamazoo River approximately 3.5 miles upstream of the former Plainwell Dam in the city of Plainwell and Gun Plain Township, Allegan County. It consists of four separate remnant structures —a waste gate structure (located approximately one mile downstream of the project area, a right diversion structure, a left diversion structure, and a head gate structure — all of which were initially constructed in 1856 by the Plainwell Water Power Company. Earthen embankments, approximately 2,520 feet in total length, are also present to connect the two diversion structures.

According to the MDNRE, the dam and associated structures were partially removed in the early 1980s such that there is no longer any "significant amount of water" impounded in the area. The primary continuing purpose of the remaining structures is to maintain flow through the mill race/power canal, which along with the Kalamazoo River, encircles the city of Plainwell and gives it the name "Island City." State and local officials have expressed intentions to leave the Plainwell No. 2 Dam structures in place to continue to provide flow through the mill race and preserve the character of the city.

The environmental questions being asked: Will the excavation of sediments from the banks and floodplains of the Kalamazoo River and the Plainwell Impoundment & #2 Dam (1) result in statically lower PCB levels in the fish and animals that inhabit this portion of the Kalamazoo River and (2) eliminate the imminent and substantial danger to both human and ecological receptors?

A synopsis of information from previous site activities: During the 1993 and 1994 Remedial Investigation/Feasibility Study, the environmental consulting firm known as Blasland, Bouck, and Lee, Inc. (BBL) collected 125 sediment samples and 135 floodplain soil samples from within the channel of the former Plainwell Impoundment and its floodplain. The total PCB concentrations of the sediment samples ranged from non-detect to 139 mg/kg, while the total PCB concentrations for the soil samples ranged from non-detect to 85 mg/kg.

In 2001, U.S. EPA conducted a two-phase sampling program in which it collected a total of 213 sediment samples and 759 soil samples from the Kalamazoo River and its floodplain. The total PCB concentrations of the sediments ranged from non-detect to 33 mg/kg for Phase I and from non-detect to 4.2 mg/kg for Phase II. The total PCB concentrations of the soil samples ranged from non-detect to 84 mg/kg for Phase I and from non-detect to 158 mg/kg for Phase II.

In 2006, BBL collected 222 sediment samples from areas in and around the Kalamazoo River that were judged to be "hotspots." The total PCB concentrations ranged from non-detect to 220 mg/kg.

In 2008, U.S. EPA collected 198 river sediment and 53 oxbow sediment samples. The total PCB concentrations from non-detect to 100mg/kg. U.S. EPA also collected 302 floodplain soil samples from 95 locations and 265 bank soil samples from 78 locations. The total PCB concentrations ranged from non-detect to 60mg/kg.

The possible classes of contaminants and the affected matrices: PCBs in river sediment and floodplain soil.

The rationale for inclusion of chemical and nonchemical analyses: Past sampling from the Kalamazoo River and within the former Plainwell Impoundment and Plainwell #2 Dam area has shown the presence of PCBs in the river sediment and floodplain soil.

Project decision conditions (If..., then...@ statements): If any of the confirmation sample results are above 5 mg/kg (commercial) or 4 mg/kg (residential) for total PCBs, then further removal of the river sediment or floodplain soil is required.

QAPP Worksheet #11
Project Quality Objectives/Systematic Planning Process Statements

Who will use the data? U.S. EPA Region V and the MDNRE.

What will the data be used for? U.S. EPA Region V and the MDNRE will use the data obtained during the project (i.e., sediment, soil, and water analytical results for total PCB content) to determine if the PRP removal contractor (Terra) has excavated enough river sediment and/or floodplain soil so that the PCB levels in the river sediments and floodplain soils are below 4 mg/kg for residential areas and below 5mg/kg, for commercial areas.

What types of data are needed (matrix, target analytes, analytical groups, field screening, off-site laboratory techniques, sampling techniques)? The analytical data needed are for total PCBs in river sediment, floodplain soils, and water samples (water column in the Kalamazoo River and water treatment samples). All START samples will be split samples collected from the same material collected by the PRP oversight contractor (Arcadis). All samples will be analyzed at a private off-site laboratory, using U.S. EPA Method 8082 for the sediment and soil samples and U.S. EPA Method 608 for the water samples.

Matrix: Sediment, soil, and water.

How "good" do the data need to be in order to support the environmental decision? The QC criteria of the selected analytical method must be met or the data qualified appropriately if the QC criteria are not met.

How much data are needed (number of samples for each analytical group, matrix, and concentration)? In general, the number of split samples collected by START will be determined by the total number of samples that Arcadis collects during the entire project. Specifically, START will collect one in every ten sediment/soil samples that Arcadis collects during the entire project, and will collect water samples (water column and water treatment samples) when new water treatment systems become operational or when significant rain events impact the current of the river. Additional samples may also be collected depending on unanticipated site conditions or at the direction of the OSC. Additionally, START will collect all applicable QC samples (duplicates and MS/MSDs), and will hand-deliver all samples to the laboratory for analysis.

Where, when, and how should the data be collected/generated? For each sediment and soil sample, START will collect a split sediment or soil sample from Arcadis, and will place this split sample material into a 4-ounce glass jar. For each water sample, START will collect a split sample from Arcadis, and will place this split water sample into two one-liter amber glass bottles. START will collect one in every ten sediment or soil samples from Arcadis, and will collect split water samples from Arcadis when new water treatment systems become operational or when site conditions most warrant the collection of a split water sample (e.g., after a storm event).

Who will collect and generate the data? WESTON START

How will the data be reported and archived? The data will be submitted electronically, and WESTON will maintain a copy of all site-related data and files for a period of 10 years in accordance with its policies. In addition, WESTON will give a copy of all data to the OSC, who will archive the data in the U.S. EPA's records center.

QAPP Worksheet #12A
Measurement Performance Criteria Table – Soil/Sediment

Matrix	Soil/Sediment
Analytical Group[1]	PCBs
Concentration Level	Low/Medium

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
S-1	U.S. EPA Method 8082 / GR-03-128	Precision—Field	RPD 50%	Field duplicate samples	S&A
		Precision – Lab	20%	Laboratory Duplicates	A
		Accuracy/Bias	Varies – See Worksheet # 28	Matrix spike and matrix spike duplicates	A
		Accuracy/Bias	Varies – See Worksheet # 28	Laboratory Control Sample	A
		Accuracy/Bias Contamination	Less than Level of Detection	Method blanks, Instrument Blanks	A
		Representativeness	NA	Adherence to soil/sediment sampling SOP and sampling plan for collection procedures and quantity of samples to collect	S
		Sensitivity	± 40 % at Quantitation Limit	Laboratory Fortified Blank at Quantitation Limit	A
		Completeness	90% of samples collected and analytical data received	Project manager assesses completeness of samples collected; laboratory project manager assesses completeness of analytical requirements per the QAPP	S&A

[1]If information varies within an analytical group, separate by individual analyte.
[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #12B
Measurement Performance Criteria Table – Water

Matrix	Water
Analytical Group[1]	PCBs
Concentration Level	Low/Medium

Sampling Procedure[2]	Analytical Method/SOP[3]	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and / or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
F-16	U.S. EPA Method 608/GR-03-128	Precision – Field	RPD 50%	Field duplicate samples	S&A
		Precision-Lab	26% -PCB 1248 42% -PCB 1254 20% for all other PCBs	Laboratory Duplicates	A
		Accuracy/Bias	Varies – See Worksheet # 28	Matrix spike and matrix spike duplicates	A
		Accuracy/Bias	Varies – See Worksheet # 28	Laboratory Control Sample	A
		Accuracy/Bias Contamination	Less than Level of Detection	Method blanks, Instrument Blanks	A
		Representativeness	NA	Adherence to sampling SOP and sampling plan for collection procedures and quantity of samples to collect	S
		Sensitivity	± 40 % at Quantitation Limit	Laboratory Fortified Blank at Quantitation Limit	A
		Completeness	90% of samples collected and analytical data received	Project manager assesses completeness of samples collected; laboratory project manager assesses completeness of analytical requirements per the QAPP	S&A

[1]If information varies within an analytical group, separate by individual analyte.
[2]Reference number from QAPP Worksheet #21 (see Section 3.1.2).
[3]Reference number from QAPP Worksheet #23 (see Section 3.2).

QAPP Worksheet #13
Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
BBL produced data from RI/FS in 1993 and 1994	Arcadis	Arcadis	Data will be used to assess sampling design rationale to help focus the sediment and floodplain soil removal	Data will be used to support final remedial approach. Data quality and DQOs associated with the data sets are unknown.
U.S. EPA produced data from 2001 sampling	U.S. EPA	U.S. EPA	Data will be used to assess sampling design rationale to help focus the sediment and floodplain soil removal	Data will be used to support final remedial approach. Data quality and DQOs associated with the data sets are unknown.
BBL produced data from 2006 sampling	Arcadis	Arcadis	Data will be used to assess sampling design rationale to help focus the sediment and floodplain soil removal	Data will be used to support final remedial approach. Data quality and DQOs associated with the data sets are unknown.
U.S. EPA produced data from 2008 sampling	U.S. EPA	U.S. EPA	Data will be used to assess sampling design rationale to help focus the sediment and floodplain soil removal	Data will be used to support final remedial approach. Data quality and DQOs associated with the data sets are unknown.

BBL – Blasland, Bouck and Lee, Inc.
RI/FS – Remedial Investigation/Feasibility Study

QAPP Worksheet #14
Summary of Project Tasks

Sampling Tasks:
1. Collection of split sediment, soil, and water samples.
2. Collection of additional samples as directed by the OSC.
3. Documentation of the details of sample locations, depths, collection dates, and collection times for each sediment, soil, or water sample.

Analysis Tasks:
1. A private laboratory (TriMatrix Laboratories, Inc. of Grand Rapids, Michigan) will prepare and process the sediment and soil samples in conjunction with U.S. EPA Method 8082, and will prepare and process the water samples in conjunction with U.S. EPA Method 608.

Quality Control Tasks:
1. The site leader/alternate site leader will evaluate all samples and applicable field quality control samples for acceptability for transport/submission to the laboratory.
2. The site leader/ alternate site leader will Implement SOPs for sample collection, packaging, transport, and storage prior to analysis.
3. The site leader/ alternate site leader will collect field duplicates and MS/MSDs per the QAPP and the sampling plan.
4. Laboratory to perform laboratory QC procedures per their SOP and this QAPP. QC procedures include analyzing method blanks, laboratory control samples and matrix spike/matrix spike duplicate samples.

Secondary Data: Not Applicable.

Documentation and Records:
1. The field team will maintain all records during sample collection and preparation for transport to the laboratory.
2. The field team will document the location and collection times and dates for all sediment, soil, and water samples in the field logbook and on the COC forms. A copy of all finalized documents and analytical data will be retained in the site files.

Data and Document Management Tasks:
The field and laboratory data (electronic and hard copy) generated during this project will be retained at the U.S. EPA Region V Regional Office. Field logbooks, sample records, and COC forms will be kept for a period of 10 years.

Data Review Tasks:
The laboratory will review all analytical data for completeness and quality prior to submitting a final data package to WESTON. This will be accomplished in accordance with the laboratories quality management plan and internal policies. A case narrative describing any quality control issues with the analyses will be submitted with the final data report. In addition, the laboratory will qualify data in accordance with its quality policies.

The WESTON Data Validator or qualified alternate (Lisa Graczyk or Tonya Balla) will validate the final data package after receipt from the laboratory. The data validation will be conducted in general accordance with the *U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* dated June 2008, along with the laboratory specific SOPs, and guidance provided in this project specific QAPP. Lastly, prior to using the analytical data in any report, WESTON will provide a data compliance check of all of the received data.

Assessment/Audit Tasks:
Assessment of field activities will be carried out by the Project Manager through daily contact with the site leader. Audits will be carried out as directed and approved by the OSC.

QAPP Worksheet #15A
Reference Limits and Evaluation Table – Soil/Sediment

Matrix: Soil/Sediment
Analytical Group: PCBs (as Aroclor); U.S. EPA 8082
SOP Reference: GR-03-128
Concentration Level: Low/Medium

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit Goal (applicable units)	Analytical Method[1]		Achievable Laboratory Limits[2]	
				MDLs	Method QLs	MDLs	QLs
PCBs		5 mg/kg					
Aroclor® 1016	12674-11-2		0.33 mg/kg	0.0070 mg/kg	0.33 mg/kg	0.0070 mg/kg	0.33 mg/kg
Aroclor® 1221	11104-28-2		0.33 mg/kg	0.011 mg/kg	0.33 mg/kg	0.011 mg/kg	0.33 mg/kg
Aroclor® 1232	11141-16-5		0.33 mg/kg	0.011 mg/kg	0.33 mg/kg	0.011 mg/kg	0.33 mg/kg
Aroclor® 1242	53469-21-9		0.33 mg/kg	0.0053 mg/kg	0.33 mg/kg	0.0053 mg/kg	0.33 mg/kg
Aroclor® 1248	12672-29-6		0.33 mg/kg	0.014 mg/kg	0.33 mg/kg	0.014 mg/kg	0.33 mg/kg
Aroclor® 1254	11097-69-1		0.33 mg/kg	0.0078 mg/kg	0.33 mg/kg	0.0078 mg/kg	0.33 mg/kg
Aroclor® 1260	11096-82-5		0.33 mg/kg	0.0056 mg/kg	0.33 mg/kg	0.0056 mg/kg	0.33 mg/kg

[1]Analytical MDLs and QLs are those documented in validated methods.

[2]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

QAPP Worksheet #15B
Reference Limits and Evaluation Table – Water

Matrix: Water
Analytical Group: PCBs (as Aroclor); U.S. EPA 608
SOP Reference: GR-03-128
Concentration Level: Low

Analyte	CAS Number	Project Action Limit (applicable units)	Project Quantitation Limit Goal (applicable units)	Analytical Method[1]		Achievable Laboratory Limits[2]	
				MDLs	Method QLs	MDLs	QLs
PCBs		N/A					
Aroclor® 1016	12674-11-2		0.20 µg/L	0.023 µg/L	0.20 µg/L	0.023 µg/L	0.20 µg/L
Aroclor® 1221	11104-28-2		0.20 µg/L	0.081 µg/L	0.20 µg/L	0.081 µg/L	0.20 µg/L
Aroclor® 1232	11141-16-5		0.20 µg/L	0.028 µg/L	0.20 µg/L	0.028 µg/L	0.20 µg/L
Aroclor® 1242	53469-21-9		0.20 µg/L	0.024 µg/L	0.20 µg/L	0.024 µg/L	0.20 µg/L
Aroclor® 1248	12672-29-6		0.20 µg/L	0.030 µg/L	0.20 µg/L	0.030 µg/L	0.20 µg/L
Aroclor® 1254	11097-69-1		0.20 µg/L	0.023 µg/L	0.20 µg/L	0.023 µg/L	0.20 µg/L
Aroclor® 1260	11096-82-5		0.20 µg/L	0.020 µg/L	0.20 µg/L	0.020 µg/L	0.20 µg/L
Total Aroclors (PCBs)	1336-36-3		0.20 µg/L	0.20 µg/L	0.20 µg/L	0.20 µg/L	0.20 µg/L

[1]Analytical MDLs and QLs are those documented in validated methods.

[2]Achievable MDLs and QLs are limits that an individual laboratory can achieve when performing a specific analytical method.

QAPP Worksheet #16
Project Schedule/Timeline Table

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Removal Action (2007)	Arcadis and Terra	May 30, 2007	December 21, 2007	N/A	N/A
First Site Mobilization (2007)	U.S. EPA and WESTON	June 4, 2007	June 7, 2007	N/A	N/A
Pollution Report Preparation	U.S. EPA	June 7, 2007	Final Nov. 2010	Pollution Reports	N/A
SAP Preparation	WESTON	June 13, 2007	June 28, 2007	SAP	June 28, 2007
Split Sampling of Sediment, Soil, and Water Samples	WESTON, MDNRE, and Arcadis	June 26, 2007	Oct. 2010	N/A	N/A
Sample Analysis	Trimatrix Laboratory	June 27, 2007	October 2010	N/A	N/A
Data Handling/Validation	WESTON	September 19, 2007	November 2010	Data Validation Packets	N/A
Second Site Mobilization (2008)	U.S. EPA and WESTON	March 5, 2008	March 7, 2008	N/A	N/A
Removal Action (2008)	Arcadis and Terra	May 12, 2008	December 10, 2008	N/A	N/A
QAPP Preparation	WESTON	August 4, 2008	May 22, 2010	QAPP	N/A
Removal Action (2009)	Arcadis and Terra	August 4, 2009	November 25, 2009	N/A	N/A
First Site Demobilization (2009)	U.S. EPA and WESTON	September 21, 2009	November 6, 2009	N/A	N/A
Second QAPP Preparation (Plainwell No. 2 Dam)	WESTON	September 30, 2009	May 2010	QAPP	N/A
Removal Action (2010)	Arcadis and Terra	May 2010	October 2010	N/A	N/A
Second Site Demobilization (2010)	U.S. EPA and WESTON	September 2010	October 2010	N/A	N/A
Final Report Preparation	WESTON	September 2010	December 2010	Final Report	December 2010

Notes:
TBD – To Be Determined

QAPP Worksheet #17
Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach):
Given that the removal is funded by the PRP, and that the PRP will be collecting the cleanup verification sediment, soil, and water samples, WESTON will collect split samples from the PRP (one in every ten sediment or soil samples) in order to confirm the accuracy of the PRP PCB analytical and monitoring results.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency (including seasonal considerations) [May refer to map or Worksheet #18 for details]:

In general, the number of split samples collected by START will be determined by the total number of samples that Arcadis collects during the entire project. Specifically, START will collect one in every ten sediment/soil samples that Arcadis collects during the entire project, and will collect water samples (water column and water treatment samples) when new water treatment systems become operational or when significant rain events impact the current of the river. Additional samples may also be collected at the direction of the OSC. Additionally, START will collect all applicable QC samples (duplicates and MS/MSDs), and will hand-deliver all samples to the laboratory for analysis.

START will not physically collect each sample, but will provide suitable sample containers (see table shown below) into which Arcadis will place the split sample material. Following the collection of the samples, START will prepare a COC for the samples, package the samples for delivery to the laboratory, and will hand-deliver the samples to Trimatrix Laboratories, Inc., of Grand Rapids, Michigan.

Sampling and Analysis Summary

Matrix	Analytical Parameter	Analytical Method (SW-846)	Containers (Numbers, Size, and Type)	No. of Sampling Locations	No. of Field Duplicate Pairs	No. of MS/MSD Pairs	Total No. of Samples to Lab[1]
Sediment/ Soil	Total PCBs	8082	One 4-oz glass jar with Teflon®-lined lid	16	2	1	18
Water	Total PCBs	608	Two 1-liter amber glass bottles with Teflon®-lined lid	5	1	0	5

[1] Total number of samples to the laboratory does not include MS/MSD samples. Note that MS/MSD or spike/duplicate analysis may require additional sample volume for water samples.

Worksheet #18
Sampling Locations and Methods/SOP Requirements Table

Sampling Location / ID Number	**Matrix**	**Depth (in.)**	**Analytical Group**	**Concentration Level**	**Number of Samples** (identify field duplicates)	**Sampling SOP Reference**[1]	**Rationale for Sampling Location**
Determined by the areas dredged by the PRP contractors	Sediment/ Soil	3-6 in.	PCBs (as Aroclors)	Low	18	F-4, F-5	See Worksheet #17
Determined by the areas dredged by the PRP contractors	Water	N/A	PCBs (as Aroclors)	Low	6	F-1, F-3	See Worksheet #17

[1]Specify the appropriate letter or number from the Project Sampling SOP References table (Worksheet #21).

Worksheet #19
Analytical SOP Requirements Table

Matrix	Analytical Group	Concentration Level	Analytical and Preparation Method / SOP Reference[1]	Sample Size	Containers (number, size, and type)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time (preparation / analysis)
Sediment/Soil	PCBs (as Aroclors)	Low	U.S. EPA 3510B/8082	115 grams	One 4-ounce wide-mouth glass jar; Teflon® -lined lid	Cool, 4°C, dark	14/40 days
Water	PCBs (as Aroclors)	Low	U.S. EPA 3510B/608	2,000 milliliters	Two 1-Liter amber glass jar; Teflon® -lined lid	Cool, 4°C, dark	7/40 days

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

Worksheet #20
Field Quality Control Sample Summary Table

Matrix	Analytical Group	Conc. Level	Analytical and Preparation SOP Reference[1]	No. of Sampling Locations[2]	No. of Field Duplicate Pairs	No. of MS/MSD	No. of Field Blanks	No. of Equip. Blanks	No. of PT Samples	Total No. of Samples to Lab
Sediment/ Soil	PCBs (as Aroclors)	Low	U.S. EPA 3510B/8082	16	2	1	N/A	N/A	N/A	18
Water	PCBs (as Aroclors)	Low	U.S. EPA 3510B/608	5	1	0	N/A	N/A	N/A	6

Notes:

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

[2]If samples will be collected at different depths at the same location, count each discrete sampling depth as a separate sampling location or station.

N/A – Not Applicable

Worksheet #21
Project Sampling SOP References Table

Reference Number	Title, Revision Date and / or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
F-1	PRP Oversight Contractor (Arcadis) Field Sampling Plan (FSP)-Water Column Sampling Procedures.	Arcadis	Boat and motor, 2 one-liter amber bottles, ISCO 3710 water sampling device, appropriate H and S equipment for boat work (PFD), and disposable gloves	N	Describes water column sampling procedures and the required equipment
F-16	Arcadis FSP-Effluent Water Grab Sampling Procedures	Arcadis	2 one-liter amber bottles and disposable gloves. Note: Arcadis will collect the sample directly from the effluent pipe.	N	Describes water quality measurement procedures and the required equipment
F-4	Arcadis FSP-Sediment Sampling Procedures.	Arcadis	GPS device, Lexan tubing and caps, hacksaw and blades, permanent markers, pie tins, disposable gloves, and sample jars.	N	Describes the procedures for sediment sampling and the required equipment.
F-5	Arcadis FSP-Soil Sampling Procedures	Arcadis	GPS device, pie tines, permanent marker, disposable gloves, and sample jars.	N	Describes the procedures for soil sampling and the required equipment

Worksheet #22
Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maint. Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference[1]
MIE pDR-1000AN	Zero in Zero Air calibration Bag	Daily 9-Volt battery replacement	Test dust levels in the air during excavation activities	Daily check of the remaining battery charge	Daily	pDR should read zero after calibration	If pDR calibration reading is greater than zero, contact manufacturer.	Site Leader and Alternate Site Leader	F-4 and F-5

[1]Specify the appropriate reference letter or number from the Project Sampling SOP References table (Worksheet #21).

QAPP Worksheet #23
Analytical SOP References Table

Reference Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
GR-09-108	Extraction of Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) from Soil, Sludge, and Wipe Samples, 2/26/09, rev.4.3, U.S. EPA 3550C	Definitive	PCBs	N/A	Trimatrix Laboratories, Inc.	N
GR-09-107	Extraction of Organochlorine Pesticides, PCBs, and Chlorinated Hydrocarbons from Water, 7/27/09, rev.5.2, U.S. EPA 608	Definitive	PCBs	N/A	Trimatrix Laboratories, Inc.	N
GR-03-128	PCBs by Gas Chromatography, 08/21/09, rev.2.5, U.S. EPA 8082	Definitive	PCBs	Agilent GC 6890 (ECD-ECD)	Trimatrix Laboratories, Inc.	N
GR-09-109	Sulfur Cleanup, 2/27/09, rev. 3.3, U.S. EPA 3660B	Definitive	PCBs	N/A	Trimatrix Laboratories, Inc.	N
GR-09-110	Sulfur Acid Cleanup, 2/15/08, rev. 3.1, U.S. EPA 3665A	Definitive	PCBs	N/A	Trimatrix Laboratories, Inc.	N
GR-10-104	Chain-of-Custody (COC), 11/30/06, rev. 2.2	Definitive	PCBs	N/A	Trimatrix Laboratories, Inc.	N

QAPP Worksheet #24
Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1]
Agilent GC 6890 (ECD-ECD)	See GR-03-128	Initial Calibration after initial setup prior to sample analysis, and when continuing calibration verification criteria not met	PCB-1016/1260 initial RSD≤ 10% or R2>0.995; SCV± 25% Continuing calibration % Drift/ % Difference ≤15%	Perform any necessary instrument maintenance and repeat initial calibration. Reanalyze all samples with failed calibration verification.	GC Analyst	GR-03-128

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

QAPP Worksheet #25
Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Agilent GC 6890 (ECD-ECD)	Parameter Set up; Tune Check	Replace septum, replace inlet liner, clip column, bake out instrument, recondition column	Check connections, replace disposables, bake out instrument, recondition column and perform leak checks	See GR-03-128	See GR-03-128	Inspect system; correct problem; re-run calibration and affected samples	GC Analyst	GR-03-128

[1]Specify the appropriate reference letter or number from the Analytical SOP References table (Worksheet #23).

QAPP Worksheet #26
Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Site Leader or Alternate Site Leader (WESTON-START)
Sample Packaging (Personnel/Organization): Site Leader or Alternate Site Leader (WESTON-START)
Coordination of Shipment (Personnel/Organization): NA -- Site Leader or Alternate Site Lead will hand-deliver samples to the laboratory
Type of Shipment/Carrier: Hand-delivery to laboratory
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Receiving Clerk, TriMatrix Laboratories, Inc., Grand Rapids, MI
Sample Custody and Storage (Personnel/Organization): Sample Receiving Clerk, TriMatrix Laboratories, Inc., Grand Rapids, MI
Sample Preparation (Personnel/Organization):Laboratory Staff, TriMatrix Laboratories, Inc., Grand Rapids, MI
Sample Determinative Analysis (Personnel/Organization): Laboratory Staff, Trimatrix Laboratories, Inc., Grand Rapids, MI
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): TBD by OSC or WESTON PM or Site Leader
Sample Extract/Digestate Storage (No. of days from extraction/digestion): Unknown
Biological Sample Storage (No. of days from sample collection): N/A
SAMPLE DISPOSAL
Personnel/Organization: Sample Receiving Clerk, TriMatrix Laboratories, Inc., Grand Rapids, MI
Number of Days from Analysis: Unknown

Worksheet #27
Sample Custody Requirements Table

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory): A COC form will be maintained from the time the sample is collected until its delivery to Trimatrix Laboratory. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, a COC form will be filled out for each sample at each sampling location utilizing the laboratory-supplied COC forms. Each individual in possession of the samples will sign and date the COC form. Each time the samples are transferred, the signatures of the persons relinquishing and receiving the samples, as well as the date and time, will be documented. A copy of the COC will be retained by the site leader or alternate site leader for the site file. When samples (or groups of samples) are not under direct control of the individual responsible for them, they will be stored in a locked container. The COC record will be considered completed when the samples are received at Trimatrix Laboratories, Inc., in Grand Rapids, Michigan. The COC record will include (at minimum) the following:

- Client Name (U.S. EPA/Weston Solutions, Inc.)
- Project Name (Allied Paper)
- Address to which Trimatrix will send the analytical results, if required to do so by mail (Weston Solutions, Inc., 20 N. Wacker Drive, Suite 1210, Chicago, IL 60606 Attn: Lisa Graczyk
- Name and Phone number of the project Laboratory Coordinator (Lisa Graczyk/Telephone No. : 312-424-3339)
- Sample ID, Sample Date, and Sample Time
- Indication of Composite or Grab Sample
- Type (s) of analysis(es) to be performed

A separate COC form will accompany each cooler in each shipment. Within the laboratory, the person responsible for sample receipt will sign and date the COC form; compare samples received against those listed on the COC form; examine all samples for possible shipping damage, leakage, and improper sample preservation; note on the COC record or laboratory receiving documentation that specific samples were damaged; notify sampling personnel as soon as possible so that appropriate samples may be re-sampled; verify that sample holding times have not been exceeded; maintain laboratory COC documentation; and place the samples in appropriate laboratory storage. If requested, the laboratory may submit internal COC documentation with the data package. Final sample disposition is completed according to laboratory license requirements.

Sample Identification Procedures:

All samples for analysis, including QC samples, will be given a unique sample number. The sample numbers will be recorded in the field logbook and on the COC paperwork, and on the shipment documents (if shipment rather than hand-delivery is required or is necessary).

START will assign each sample a unique project sample number. The project sample number highlights the suspected contaminated area and location, and will be used for documentation purposes in field logbooks, as well as for presentation of the analytical data in memoranda and reports. The project sample numbering system will be composed of the components below.

Project Identifier

The first part of the project sample numbering system will be the three-character designation PD2. PD2 corresponds to Plainwell Dam #2 site.

Sample Date

This shall consist of a six digit date (i.e., 091309) for September 13, 2009.

Sequence Identifier

This shall consist of the following:

- A two-digit sequence number that tracks the number of samples collected from the Site. Sequence 01 refers to the first sample, and sequence 02 refers to the second sample.
- A two-letter designation will be used to differentiate between a sediment sample and a water sample. SD will stand for a sediment sample, while WT will stand for a water sample.
- If the sample is a field duplicate sample, the above will be combined with DP.
- The Arcadis-designated sample name will then be added to the end of the START-designated name.
- Field duplicate samples will be submitted to the laboratory without reference (i.e., the laboratory will not be informed that the sample is duplicate).

Some examples of the START project sample numbering system are as follows:

- PD2-091309-01-SD-DP/K55025: Plainwell Dam #2 site; sediment sample collected on September 13, 2009; duplicate of the first sample collected at this Site; Arcadis sample name is K55025.
- PD2-091309-WT-02/W_SA1N_EffluB_0002: Plainwell Dam #2 Site; water treatment sample collected on September 13, 2009; second water sample collected at this site; Arcadis sample name is W_SA1N_EffluB_0002.

QAPP Worksheet #28
QC Samples Table
TriMatrix Laboratories, Inc., Grand Rapids, Michigan

Worksheet:	28A	**Concentration Level**	Low
Analytical Group:	U.S. EPA 608	**Samplers Name**	Michael Browning/Jay Rauh
Analytical SOP Reference:	GR-03-128	**Field Sampling Organization**	Weston Solutions, Inc.
Matrix:	Liquids	**Analytical Organization**	TriMatrix Laboratories, Inc.
Sampling SOP	WSOP-1, 2, 4, and 7	**No. of Sample Locations**	Varies

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch	No target analyte concentrations above the reporting limit (RL).	Re-extract and re-analyze all affected samples. If insufficient volume, re-analyze only. Qualify data as needed.	Chemist	Contamination	Less than reporting limit
Surrogate Spike	Every analytical sample	Decachlorobiphenyl -30-115%R Tetrachloro-m-xylene-43-115%R	Check for matrix effects. If none, re-analyze samples or re-extract if sufficient volume and re-analyze. Qualify data as needed.	Chemist	Accuracy/Bias	Decachlorobiphenyl - 30-115%R Tetrachloro-m-xylene-43-115%R
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LSCD)	One per sample preparation batch	Refer to QC Limit Table below	If sufficient volume, re-extract and re-analyze all affected samples. If insufficient volume, re-analyze extracts. Qualify data as needed.	Chemist	Accuracy/Bias	Laboratory Control Sample/Laboratory Control Sample Duplicate RPD for each Arochlor-20% RPD
Instrument Duplicate Analysis	One per sample preparation batch	Refer to QC Limit Table below	1) Review data for usability. 2) Narrate any outliers.	Chemist	Precision-Laboratory	Aroclor 1248-26% RPD Aroclor 1254-42% RPD All other Aroclors-20% RPD

Matrix Spike	One QC pair per sample preparation batch	Refer to QC Limit Table below	Check for errors in calculations and spike preparation. Check for matrix effects. If no errors and associated LCS in control, qualify failing analytes as estimated.	Chemist	Accuracy/Bias	Aroclor 1016 -62-114%R Aroclor 1260-46-132%R See QC Limit Table for all other Aroclors
Matrix Spike Duplicate	One per sample preparation batch	Refer to QC Limit Table below	As above	Chemist	Precision-Laboratory	Aroclor 1016-10% RPD Aroclor 1260-11% RPD See QC Limit Table for all other Aroclors

QC – quality control
LCS – laboratory control samples

Worksheet:	28B	**Concentration Level**	Low/Medium
Analytical Group:	U.S. EPA 8082	**Samplers Name**	Michael Browning/Jay Rauh
Analytical SOP Reference:	GR-03-128	**Field Sampling Organization**	Weston Solutions, Inc.
Matrix:	Soils	**Analytical Organization**	TriMatrix Laboratories, Inc.
Sampling SOP	WSOP-1, 2, 4, and 7	**No. of Sample Locations**	Varies

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch	No target analyte concentrations above the reporting limit (RL).	Re-extract and re-analyze all affected samples. If insufficient volume, re-analyze only. Qualify data as needed.	Chemist	Contamination	Less than reporting limit
Surrogate	Every analytical sample	Decachlorobiphenyl -36-136% R Tetrachloro-m-xylene-46-120%R	Check for matrix effects. If none, re-analyze samples or re-extract if sufficient volume and re-analyze. Qualify data as needed.	Chemist	Accuracy/Bias	Decachlorobiphenyl - 36-136% R Tetrachloro-m-xylene-46-120%R
Laboratory Control Sample/ Laboratory Control Sample Duplicate (LCS/LSCD)	One per sample preparation batch	Refer to QC Limit Table below	If sufficient volume, re-extract and re-analyze all affected samples. If insufficient volume, re-analyze extracts. Qualify data as needed.	Chemist	Accuracy/Bias	Laboratory Control Sample/Laboratory Control Sample Duplicate RPD for each Arochlor-20% RPD
Instrument Duplicate Analysis	One per sample preparation batch	Refer to QC Limit Table below	1) Review data for usability. 2) Narrate any outliers.	Chemist	Precision-Laboratory	20% RPD
Matrix Spike	One QC pair per sample preparation batch	Refer to QC Limit Table below	Check for errors in calculations and spike preparation. Check for matrix effects. If no errors and associated LCS in control, qualify failing analytes as estimated.	Chemist	Accuracy/Bias	Aroclor 1016 -48-126%R Aroclor 1260-52-136%R See QC Limit Table for all other Aroclors
Matrix Spike Duplicate	One per sample preparation batch	Refer to QC Limit Table below	As above	Chemist	Precision-Laboratory	20% RPD

QAPP Worksheet #28
QC Samples Table (Continued)
TriMatrix Laboratories, Inc., Grand Rapids, Michigan

Liquid Samples	LCS/LCSD Control Limits					MS/MSD Control Limits				
Compound			%R LCL	%R UCL	RPD			%R LCL	%R UCL	RPD
Aroclor-1016			58	109	20			62	114	10
Aroclor-1221			68	121	20			70	130	20
Aroclor-1232			72	123	20			70	130	20
Aroclor-1242			54	113	20			37	141	40
Aroclor-1248			49	139	20			12	125	20
Aroclor-1254			66	126	20			60	117	17
Aroclor-1260			65	114	20			46	132	11

Soil Samples										
Compound			%R LCL	%R UCL	RPD			%R LCL	%R UCL	RPD
Aroclor-1016			72	117	20			48	126	20
Aroclor-1221			58	147	20			54	157	20
Aroclor-1232			85	125	20			68	130	20
Aroclor-1242			73	118	20			40	135	20
Aroclor-1248			67	131	20			39	140	20
Aroclor-1254			73	125	20			56	138	20
Aroclor-1260			77	123	20			52	136	20

QAPP Worksheet #29
Project Documents and Records Table

Sample Collection Documents and Records	On-Site Analysis Documents and Records	Off-Site Analysis Documents and Records	Data Assessment Documents and Records	Other
Field Notes	N/A	Sample Receipt, Custody, and Tracking Records	Field Sampling Audit Checklist	Investigation Summary Report
Chain-of-Custody Records		Standard Traceability Logs	Field Analysis Audit Checklist	
Air Bills		Equipment Calibration Logs	Fixed Laboratory Audit Checklists	
Custody Seals		Sample Prep Logs	Data Validation Reports	
Telephone Logs		Run Logs	Corrective Action Forms	
Corrective Action Forms		Equipment Maintenance, Testing, and Inspection Logs	Telephone Logs	
Photos		Corrective Action Forms		
Field Diagrams		Reported Field Sample Results		
		Reported Results for Standards, QC Checks, and QC Samples		

QAPP Worksheet #30
Analytical Services Table

Matrix	Analytical Group	Concentration Level	Sample Locations/ID Number	Analytical SOP	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Soil	PCBs	Low	See the map	PCBs in Soil-USEPA 8082	24-hour turnaround for results/28 days for full data package	TriMatrix Laboratories, Inc., 5560 Corporate Exchange Court, SE, Grand Rapids, Michigan 49512, Lisa Harvey: Project Manager, Telephone: (616) 975-4500	N/A
Water	PCBs	Low	Determined by Arcadis	PCBs in Water-USEPA 608	24-hour turnaround for results/28 days for full data package	TriMatrix Laboratories, Inc., 5560 Corporate Exchange Court, SE, Grand Rapids, Michigan 49512, Lisa Harvey: Project Manager, Telephone: (616) 975-4500	N/A

QAPP Worksheet #31
Planned Project Assessments Table

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (title and organizational affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Lab Audit	Once during sample analyses	External	U.S. EPA or their designated contractor	Unknown, U.S. EPA	QC Manager, TriMatrix Laboratories, Inc.	QC Manager, TriMatrix Laboratories, Inc.	QC Manager, TriMatrix Laboratories, Inc.
Field Audit	Once during onsite field work	Internal	Weston Solutions, Inc.	Chris Lantinga or his designee, Project Manager WESTON	Michael Browning, Site Lead, Dynamac START	Mike Browning, Site Lead, Dynamac START	Mike Browning, Site Lead, Dynamac START

QAPP Worksheet #32
Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Health and Safety Audit	Completed Form	(1)Michael Browning, Site Leader, DYNAMAC START (2)Jay Rauh, Alternate Site Leader, WESTON START (3)Tonya Balla, Health and Safety Officer, WESTON START	One Day	Provide corrective action documentation in writing	(1)Chris Lantinga, Project Manager, WESTON START (2)Tonya Balla, Health and Safety Officer, WESTON START	Two Days
Lab Audit	Written Report	(1)Lisa Graczyk, Laboratory Coordinator, WESTON START (2)Chris Lantinga, Project Manager, WESTON START (3)Lisa Harvey, Project Manager, Trimatrix Laboratories	One Day	Provide corrective action documentation in writing	(1)Lisa Graczyk, Laboratory Coordinator/QA officer, WESTON START (2)Chris Lantinga, Project Manager, WESTON START	Seven Days
Field Audit	Written Report	(1)Michael Browning, Site Leader, DYNAMAC START (2)Jay Rauh, Alternate Site Leader, WESTON START	One Day	Obtain documentation of corrective action from Field Team Members	(1)Chris Lantinga, Project Manager, WESTON START (2)Michael Browning, Site Leader, DYNAMAC START (3)Jay Rauh, Alternate Site Leader, WESTON START	Two Days

QAPP Worksheet #33
QA Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Data Validation Report	To be prepared following receipt of an analytical data package	Two weeks following receipt of final data package from laboratory	Lisa Graczyk, Laboratory Coordinator, WESTON START.	Chris Lantinga, Project Manager, START
Final Project Report	To be prepared following receipt of all analytical data validation	One month following receipt of all data validation reports	Chris Lantinga, Project Manager, WESTON	Sam Borries, OSC, U.S. EPA
Monthly Report	Every month for the prior month activities	20th of month for the prior month activities	Chris Lantinga, Project Manager, WESTON	Sam Borries, OSC, U.S. EPA

QAPP Worksheet #34
Verification (Step I) Process Table

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-custody forms	The Site Leader or Alternate Site Leader will place all completed COC forms in the appropriate site file. All filed COC forms will be available to the Weston Project Manager and/or OSC, so that the Project Manager and/or OSC can review the COC forms for completeness and accuracy.	I	Michael Browning, DYNAMAC START Jay Rauh, WESTON START
Field Logbooks/Field notes	The field logbooks/field notes will be reviewed internally (by the Project Manager) and placed in the site file. A copy of the field notes will be attached to the final report.	I	Michael Browning, DYNAMAC START Jay Rauh, WESTON START Chris Lantinga, WESTON START
Laboratory data	All laboratory data will be verified by the QA officer of the laboratory performing the sample analyses. The laboratory data will be validated in accordance with the procedures described in Worksheet #s 35 and 36. WESTON will perform a compliance check of all data received from Trimatrix Laboratories.	I E	QC Manager, TriMatrix Laboratories, Inc. Lisa Graczyk, WESTON START

Notes:

COC – Chain of Custody
OSC – On Site Coordinator
QA- Quality Assurance
QC- Quality Control

QAPP Worksheet #35
Sampling and Analysis Validation (Steps IIa and IIb) Process Table

Step Iia / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	SOPs and logbook	The Project Manager will ensure that all SOPs are followed in the field through weekly conversations with the site leader and/or alternate site leader by reviewing the site logbook when the Project Manager is on site.	Chris Lantinga, WESTON START
IIa	Preliminary Data and Final Analytical Data Package	The Laboratory Coordinator will review the preliminary data and final analytical data package to ensure that WESTON received all requested analyses and to ensure that Trimatrix Laboratories has met the required project quantitation limits.	Lisa Graczyk, WESTON START
IIb	Final Analytical Data Package	The data validator will perform data validation of the final analytical data package to ensure that all QC requirements specified in the QAPP were met. The data validation will be conducted in general accordance with the *U.S. EPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, June 2008*. Note: WESTON will perform a compliance check of all U.S. EPA reviewed data.	Lisa Graczyk, WESTON START

Notes:

SOP-Standard Operating Procedure
QC-Quality Control
QAPP-Quality Assurance Project Plan
WESTON- Weston Solutions, Inc.
START – Superfund Technical Assessment and Response Team

QAPP Worksheet #36
Sampling and Analysis Validation (Steps IIa and IIb) Summary Table

Step IIa / IIb	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
IIb	Sediment/Soil	Total PCBs	Low	U. S. EPA NFG	Lisa Graczyk, Laboratory Coordinator, DYNAMAC START
IIb	Water	Total PCBs	Low	U. S. EPA NFG	Lisa Graczyk, Laboratory Coordinator, DYNAMAC START

Notes:

PCBs- -Polychlorinated Biphenyls
NFG- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Method Data Review, June 2008*
U.S. EPA – United States Environmental Projection Agency
START – Superfund Technical Assessment and Response Team
WESTON- Weston Solutions, Inc.

Data verification and validation will be completed by contractor prior to finalization of the data and release of the data set.

An abbreviated data validation review will be completed, which will include evaluation of the results for method-specific quality control analyses (e.g., results of method blanks and applicable instrument blanks, results for all applicable MS/MSDs, and LCSs analyses, and the results of all applicable laboratory duplicate and/or triplicate sample analyses) with respect to method-specific and laboratory-established control limits, as may be applicable. Instrument calibrations, calculations, and transcriptions will not be checked because the laboratories will be responsible for 100 percent verification of these results and procedures.

Data qualifiers will be applied to the results according to procedures described in the U.S. EPA Contract Laboratory Program national functional guidelines for data review (U.S. EPA 2002), as applicable, with modifications as appropriate to accommodate method-specific quality control requirements or when specific MQOs and DQIs established for this project (e.g., control limits for bias and precision) are not achieved.

Algorithms to Assess Quality Control Results

Data verification includes checking that quality control procedures were included at the required frequencies and that the quality control results meet control limits defined in the method descriptions or by the project DQIs. The equations that will be used to determine whether measurement targets for project DQIs were met for each quality control procedure are provided below.

Duplicate Analyses—Precision for duplicate chemical analyses will be calculated as the RPD between the duplicate samples. This formula will be used to assess precision for both laboratory and field duplicate samples:

$$\%RPD = \{(D_1 - D_2) : [(D_1 + D_2)/2]\} x100$$

where:

D_1 = sample value

D_2 = duplicate sample value.

QAPP Worksheet #36
Validation (Steps IIa and IIb) Summary Table (continued)

Matrix Spikes and Surrogate Recoveries—Spiked samples provide an indication of the bias of the analysis system. The recovery of matrix spikes and surrogate spikes will be calculated as the ratio of the recovered spike concentration to the known spiked quantity:

$$\%R = [(A - B)/C] \times 100$$

where:

A = the analyte concentration determined experimentally from the spiked sample

B = the background level determined by a separate analysis of the unspiked sample

C = the amount of the spike added.

Completeness—Completeness will be calculated for each sample type by dividing the number of valid measurements (all measurements except rejected data) actually obtained by the number of valid measurements that were planned:

$$\%\text{Completeness} = [\text{Valid Data Obtained/ Total Data Planned}] \times 100$$

To be considered complete, the data sets must also contain all quality control check analyses that verify the precision and accuracy of the results.

Sensitivity—The detection limit of the sample preparation and analysis process is defined as "the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte is greater than zero" (40 CFR 136B). In other words, it is the point at which qualitative, not quantitative, identification can be made. In practice, the limit of detection is defined as 3 times the standard deviation of the blank or background response adjusted for the amount of sample typically extracted and the final extract volume of the method.

Best professional judgment is used to adjust the limit of detection upward in cases where high instrument precision (i.e., low variability) results in a calculated limit of detection and equivalent instrument response less than the absolute sensitivity of the analytical instrument. The actual reporting limit for environmental samples is generally higher than the instrument detection limit because the sample matrix tends to contribute to fluctuations in the instrument's background signal. Laboratory personnel will determine reporting limits based on their experience with samples of similar matrix to those collected for this study and on the response of each instrument to samples for this study. The MRLs will be verified during data validation.

QAPP Worksheet #37
Usability Assessment

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used:

Data, whether generated in the field or by the laboratory, will be tabulated and reviewed for completeness and representativeness by the site leader or alternate site leader, and will be reviewed by the WESTON laboratory coordinator/data validator for precision and accuracy. The review of these data quality indicators (DQI) will compare the DQI with the data quality objects (DQO) detailed in the project-specific QAPP and in the utilized analytical methods.

Questions about the data, as observed during the data review process, will be resolved by contacting the site leader (and/or alternate site leader) and/or Trimatrix Laboratories, Inc. for resolution. All communications will be documented including the resolution to the observed deficiencies. Hard copies of all original data and deliverables will be kept in the Technical Direction Document file.

When the data do not meet the project DQOs, WESTON START will investigate the root cause of the deficiency. Possible reasons for these deficiencies may include laboratory operation, such as the laboratory's failure to adjust for the extraction weight on high-moisture-content soil; failure of laboratory reporting limits to meet site Action Limits; or poor correlation between field screening and laboratory results. In these situations, WESTON START will discuss corrective actions with the OSC. These actions may include:

- Re-sampling for all or some of the parameters;
- Preparing a technical memorandum for the site file that details the limitations of the data;
- Validating the data at a higher tier level to better qualify the results; and
- Preparing a technical memorandum determining the bias of field results.

Describe the evaluative procedures used to assess overall measurement error associated with the project:

The following specific items will be assessed in the manner described below:

Precision – Results of all laboratory duplicates and field duplicates will be presented in the laboratory data validation report. For each duplicate pair, the relative percent difference (RPD) will be calculated for each parameter with results greater than or equal to the quantitation limit. The RPDs will be checked against the measurement performance criteria presented on Worksheets #12A and 12B. The RPDs exceeding criteria will be identified on the tables in the final report with appropriate qualifiers. A discussion will follow that summarizes the results of the laboratory precision. Any conclusions about the precision of the analyses will be drawn and any limitations on the use of the data will be described in the final report.

Accuracy/Bias Contamination – Results for all laboratory method blanks and instrument blanks will be presented in the laboratory data validation report. The results for each parameter will be checked against the measurement performance criteria presented on Worksheets #12A and 12B. Results for parameters that exceed criteria will be identified on the tables in the final report with appropriate qualifiers. A discussion will follow summarizing the results of the laboratory accuracy/bias. Any conclusions about the accuracy/bias of the analyses based on contamination will be drawn and any limitations on the use of the data will be described in the data validation report.

Overall Accuracy/Bias – The results of the continuing calibration standards will be presented in the laboratory data validation report. These results will be compared to the requirements listed on Worksheets #12A and 12B. A discussion will follow summarizing overall accuracy/bias. Any conclusions about the overall accuracy/bias of the analyses will be drawn and any limitations on the use of the data will be described on the data validation report.

Sensitivity – All sample results will be presented in tabular format for each detected PCB parameter. The sample results for each detected parameter will be checked against the contract required quantitation limits. Results for detected parameters that do not meet the contract required quantitation limits will be discussed in the data validation report. Any conclusions about the sensitivity of the detected parameters will be drawn and any limitations on the use of the data will be described in the data validation report.

Representativeness – Representativeness will be maintained by the site leader and alternate site leader who will ensure that all sampling personnel are adhering to the sampling procedures dictated in the WESTON START sampling and analysis plan and the Arcadis field sampling plan. In addition, the project manager will be in close contact with the site leader to ensure that the site leader and alternate site leader is following proper sampling techniques. Any conclusions about the representativeness of the sampling will be drawn and any limitations on the use of the data will be described in the validation report.

Completeness – A completeness check will be done on all samples collected in the field and on the data generated by Trimatrix Laboratory. Completeness criteria are presented on Worksheets #12A and 12B. Completeness will be calculated as follows: For each collected sample, completeness will be calculated as the number of samples collected and the number of analyses performed, divided by the total number of planned sample collection points and analyses. A discussion will follow summarizing the calculation of data completeness. Any conclusions about the completeness of the data for each parameter will be drawn and any limitations on the use of the data will be described in the data validation report.

Reconciliation – Each of the project quality objectives presented on Worksheets #12A and 12B will be examined to determine if the objective was met. Each PCB parameter will first be evaluated in terms of the major impacts observed from the data validation, DQIs, and measurement performance criteria assessments. Based on the results of these assessments, the WESTON laboratory coordinator/data validator will determine the quality of the data. Based on the determined quality of the data, the WESTON laboratory coordinator/data validator will determine the usability of the data for each analysis. Based on the usability of the data from all analyses for an objective, it will be determined if the project quality objective was met. The final report will include a summary of all of the points that went into the reconciliation of each objective. As part of the reconciliation of each objective, conclusions will be drawn and any limitations on the usability of any of the data will be described in the data validation report

Identify the personnel responsible for performing the usability assessment: The site leader will determine the usability of field data. The WESTON laboratory coordinator/data validator will validate the data and will conduct a compliance check of the data to determine the usability of the analytical data. The Project Manager, Chris Lantinga, will be responsible for evaluating the overall usability of the sample data for meeting the project objectives.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies: A data validation report will be prepared by the WESTON laboratory coordinator/data validator. Overall usability of the sample data for meeting the project objectives will be described in the final report to be prepared by the Project Manager.

APPENDIX A

FIELD SAMPLING SOPs

SUPERFUND TECHNICAL ASSESSMENT RESPONSE TEAM
STANDARD OPERATING PROCEDURE

SOP 302
SURFACE SOIL SAMPLING

1.0 INTRODUCTION

The purpose of this Standard Operating Procedure (SOP) is to provide Roy F. Weston, Inc. (WESTON®), Superfund Technical Assessment Response Team (START) members with a step-by-step guide for collecting representative surface soil samples using scoops and bucket augers.

2.0 MATERIALS REQUIRED

Below is a list of the materials needed for surface soil sampling events. Both dedicated and reusable sampling equipment are required.

- Personal protective equipment (as specified in the Health and Safety Plan)
- Sampling plan
- Maps/sketches
- Compass
- Tape measure (up to 300 ft)
- Survey flags/stakes
- Aluminum homogenization pans
- Sample jars
- Logbook
- Sample labels/tags
- Chain-of-custody forms and custody seals
- Field data sheets
- Coolers
- Ice
- Decontamination equipment (brushes, buckets, garden sprayer, phosphate-free soap, water, etc.)
- Ziploc® bags
- Plastic sheeting
- Paper towels
- Ball-point pen
- Permanent marker
- Grease pencil
- Marking spray paint
- Digital camera or a camera with film
- Air monitoring equipment [Micro FID, Multi RAE 5 Gas detector, etc.]
- Plastic sample scoops, if applicable
- Bucket auger, if applicable
- Thin-walled tube sampler, if applicable

- Plastic garbage bag
- Scissors

3.0 SAFETY PRECAUTIONS

Due to unknown constituents of the soil media, the exposure potential for personnel exists and must be of primary concern. Before any soil sampling is performed, a Health and Safety Plan (HASP) must be approved by the Regional Safety Officer.

1. Follow the HASP safety schedule.
2. Determine the appropriate levels of protection to be worn by personnel.
3. Conduct air monitoring in the breathing zones and screen the sample location holes once they are selected.
4. Ensure that equipment is properly decontaminated and in working condition prior to the mobilizing to the site.
5. Coordinate efforts and staffing with the client or agency with which you are working.

4.0 SAMPLING PROCEDURES

1. Perform a general site reconnaissance to verify actual site conditions consistent with the HASP.
2. Identify and mark all sampling locations using sample flags or stakes as specified in the sampling plan. *All sample locations should be measured, documented, and mapped in reference to a permanent marker, i.e. specified utility pole, benchmark, property marker, etc.*
3. Mark the pertinent site information in a site logbook and on field data sheets. *When large amounts of samples are collected, field data sheets allow for easy organization in addition to logbook entries.*
4. Make sure all sampling equipment is properly decontaminated prior to sampling.
5. Wear clean, disposable surgical gloves for each sampling location.
6. Begin sampling by cutting or pulling back debris with a stainless steel or dedicated plastic scoop.
7. Cover the sample location area with plastic sheeting if the soil has a high probability of contamination.
8. Continue cutting to the required depth. Generally, surface sampling is considered 0-3 inches below the surface. It is recommended sample holes be kept the same size diameter (suggested 6 inches) even when using scoops to keep samples relative to each other. Sample collection will focus on soil particles, not plant and tree roots, stones, rocks, concrete and other materials intermixed in the soil matrix.
9. If a grab sample is to be collected, transfer the sample volume directly into the sample container using a sampling device. *Check the preferred sampling apparatus list for various analytical parameters.* A grab sample pertains to a discreet depth or area in a given matrix.
10. Transfer the sample volume to a homogenization container if the sample is a composite sample or a pseudo-grab sample. A composite sample is a mixture of different depths, areas, and/or strata. Composite samples are not recommended for

the collection of VOC samples because mixing causes volatile compounds to evaporate.

11. There are several homogenization techniques. The "quartering technique" requires the total volume of samples be divided into fourths inside the aluminum pan. Each quarter is then mixed individually, then the quarters are combined. This technique is repeated until a thorough mixing has occurred. The second method is the "bakers technique", which simply entails mixing the soil volume with hands covered by surgical gloves or sampling scoops. The "shake and bake technique" allows the cleanest mixing. This technique requires emptying the sample volume into a Ziploc® bag, sealing the bag, and then shaking the bag until the sample volume is thoroughly mixed. Note the qualities (color, texture, etc.) of the homogenized sample.
12. Place the sample in the designated sample container after the sample has been homogenized.
13. Label the sample container. Sample labels and tags are to be filled out with a permanent marker (*ball point pen ink bleeds when wet).* Use a grease pencil to fill out labels and tags for samples to be analyzed for VOCs. Additionally, it is recommended that the bottom of the sample jar be marked with the time of collection, the sample location, and the sampler's initials, in case the labels are rendered illegible.
14. Place the sample jar into an appropriate sized Ziploc® bag.
15. Place the sample on ice, if applicable. Generally, soil samples do not require any preservative; however, unless told otherwise, it is always good practice to put samples on ice.
16. Decontaminate the sampling apparatus using the proper procedure (see Section 6.0 Decontamination of Sampling Equipment).
17. Complete the chain-of-custody form in a clear and concise manner.
18. Repeat steps 1-17 for each sample location.

5.0 SAMPLING DEVICES

Three common sampling devices used by START personnel include the sample scoop, the auger, and the thin-walled sampler/corer. The sample scoop includes both dedicated disposable plastic scoops and stainless steel scoops. Augers include bucket augers and hand augers. The thin-walled sampler/corer is the least used device of the three.

5.1 Scoops

Scoops make sampling quick and easy. Any time rough terrain is encountered, scoops are the ideal device. Generally disposable scoops are used because no wet decontamination is required. Never reuse dedicated scoops and always make sure proper decontamination has been performed for non-disposable sample scoops.

5.2 Bucket and Hand Augers

Augers are manually driven stainless steel sampling devices. The hand auger is a smaller version of the bucket auger. Augers tend to fluff sample volumes. Because of their design, augers are recommended for composite sampling. Augers are not recommended for VOC sampling because volatiles will be driven off.

5.2.1 Auger Sampling Procedures

1. Decontaminate augers before collecting first sample.
2. Cut a 12-inch hole in the plastic sheeting around sample location using scissors.
3. Discard debris and other surface material.
4. Place the auger perpendicular to the ground and twist the "T" handle in a clockwise rotation until the desired depth is achieved. To determine the depth of the sample measure the actual removed core or the depth of the newly bore hole.
5. Retrieve the specified sample volume. Any additional sample volume can be returned to the sample hole.
6. Place the sample volume into a homogenization pan and mix thoroughly.
7. Place the sample in the designated sample container. Note: Only VOA *containers are to be packed tightly.*
8. Label the sample container. Sample labels and tags are to be filled out with a permanent marker (*ball point pen ink bleeds when wet*). Use a grease pencil to fill out labels and tags for samples to be analyzed for VOCs. Additionally, it is recommended that the bottom of the sample jar be marked with the time of collection, the sample location, and the sampler's initials, in case the labels are rendered illegible.
9. Place the sample jar into an appropriate sized Ziploc® bag.
10. Place the sample on ice, if applicable. Generally, soil samples do not require any preservative; however, unless told otherwise, it is always good practice to put samples on ice.
11. Decontaminate the auger using the proper procedure (see Section 6.0 Decontamination of Sampling Equipment).
12. Complete the chain-of-custody form in a clear and concise manner.
13. Repeat steps 1-12 for each sample location.

Note: A major drawback for auger sampling is that roots, stones and other materials will not allow for good penetration. Different sample locations may have to be selected to collect samples.

5.3 Thin-Walled Sampler/Corer

The thin-walled sampler/corer is the least used of the common sampling devices. It works similar to an auger; however, it has a much smaller diameter and the core is visible from the side of the sampler barrel. This device is even more prone to refusal than the bucket auger. This device works well in moist soils with small grain sizes.

5.3.1 Corer Sampling Procedures

1. Decontaminate the augers before collecting the first sample.
2. Cut a 12-inch hole into plastic sheeting around sample location.
3. Discard debris and other surface material.
4. Place the thin-walled sampler perpendicular to the ground and twist the "T" handle in a clockwise rotation until desired depth is achieved.
5. Retrieve the specified sample volume. Any additional sample volume can be returned to the sample hole.
6. Place the sample volume into a homogenization pan and mix thoroughly.
7. Place the sample in the designated sample container.
8. Label the sample container. Sample labels and tags are to be filled out with a permanent marker only (*ball point pen ink bleeds when wet*). Use a grease pencil to fill out labels and tags for samples to be analyzed for VOCs. Additionally, it is recommended that the bottom of the sample jar be marked with the time of collection, the sample location, and the sampler's initials, in case the labels are rendered illegible.
9. Place the sample jar in an appropriate sized Ziploc® bag.
10. Place the sample on ice, if applicable. Generally, soil samples do not require any preservative; however, unless told otherwise, it is always good practice to put samples on ice.
11. Decontaminate the auger using the proper procedure (see Section 6.0 Decontamination of Sampling Equipment).
12. Complete the chain-of-custody form.
13. Repeat steps 1-12 for each sample location.

6.0 DECONTAMINATION OF SAMPLING EQUIPMENT

This procedure is arguably the most important step in sound sample collection. Poor decontamination will result in cross-contamination and inaccurate sample results. The adequacy of the decontamination is generally tested by daily rinsate blanks. The following procedures pertain to the three sampling devices noted in this SOP.

1. Determine an area to be used as a decontamination station and lay plastic sheeting down.
2. Fill and pressurize a garden sprayer with distilled water. Fill one decontamination bucket with distilled water and Alconox®. Fill and pressurize another garden sprayer (if available) with de-ionized water for the final rinse.
3. Brush off soil residue from the sampling device with a dry brush.
4. Quickly spray the sampling device with the garden sprayer to loosen the soil before placing the sampling device into the soapy water.

5. Put the sampling device into soapy water bucket. Remove soil residue with a long-handled brush, toilet brush or cleaning device. Spray off soap residue with distilled water.
6. Place the sampling device into another bucket and spray the sampling device thoroughly again with distilled water.
7. Final rinse the sampling device with de-ionized water. If solvents or weak acids are used for the final rinse, see START SOP No. 406, Investigative Derived Waste.
8. If stainless steel scoops are used, use multiple scoops so that decontamination does not have to be after every hole.
9. Repeat steps 1-7.
10. Contact the OSC to determine if decontaminated water may be dumped on site. Be sure to address this issue before the sampling event occurs. All PPE and other refuse generated can be disposed as solid industrial waste.

7.0 REFERENCES

EPA. 1991. *Compendium of Emergency Response Team (ERT) Soil Sampling and Surface Geophysic Procedures.* Office of Solid Waste and Emergency Response, Washington, DC. EPA/540/P-91/006.

EPA 1991. *Removal Program Representative Sampling Guidance.* Volume 1 - Soil. Office of Solid Waste and Emergency Response, Washington, DC. 9630.4-10 P892-963408.

WESTON® (Roy F. Weston, Inc.) 1993. *Standard Practices Manuel for Soil Sampling With a Spade, Scoop and Stainless Surface Soil Sampler Auger and Tube Sampler.* West Chester, PA.

Attachment: 1

ATTACHMENT 1

SOIL SAMPLING DATA SHEET

Sample Number(s): ____________
Date: ____________
Time: ____________

Soil Sampling Data Sheet

Site Name: __________________ **Sampler:** __________________

Sample Depth: ____ Surface (0-0.5 ft) _____ Shallow (0.5-5.0 ft)

Sample Method(Circle One):_____Scoop (2,3,4,5,6,7,8,A,C,+) ____ Hand Auger(2,3,4,5,6,7,B,+,-)

_____Slide-Hammer (1,2,3,4,5,6,7,8,A,B,+,-) ____ Open Tube (A,+,-)

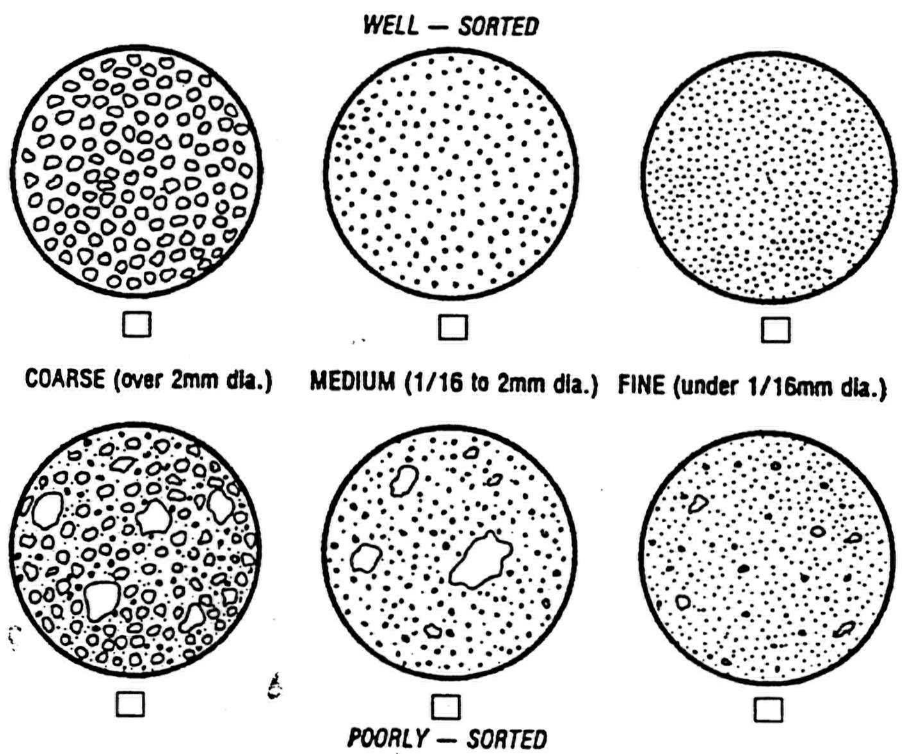
_____Split/Solid Tube (1,2,3,4,5,6,7,8,A,B,-) ____ Thin-Wall Tube(8,A,-)

Preferred Methods

1 - Volatiles	5 - PCBs	A - Grab	+ - Surface
2 - Semi-Volatiles	6 - TPH	B - Composite (Vertical)	- - Shallow
3 - Primary Metals	7 - Rad	C - composite (Areal)	
4 - Pesticides	8 - Geotechnical		

Soil Description (Munsell): Chart _____ Value _____ Hue _____

Grain Size and Distribution:



APPENDIX B

ANALYTICAL SOPs

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Sulfuric Acid Cleanup

SW-846 Method 3665A

APPROVALS:

Area Supervisor: ______________________ Date: 12/15/08
Brian J. Hall

QA Officer: ______________________ Date: 12-15-08
Tom C. Boocher

Operations Manager: ______________________ Date: 12/15/08
Jeff P. Glaser

Procedure Number: GR-09-110
Revision Number: 3.1

Date Initiated: 3/30/94
Effective Date: 12/31/08

Date Revised: 12/15/08
Pages Revised: All

By: Andrea S. Colborn
Total Number of Pages: 8

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to rigorous cleanup of polychlorinated biphenyl (PCB) hexane extracts. All PCB extracts must undergo sulfuric acid cleanup to minimize quantitative chromatographic interference.

1.2 Sulfuric acid cleanup is not applicable to cleanup of extracts for other analytes. The acid removes many organics, including some pesticides.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 1, December, 1996, Method 3665A, "Sulfuric Acid/Permanganate Cleanup"

3.0 SUMMARY OF PROCEDURE

3.1 Concentrated sulfuric acid is added to a hexane extract. The mixture is thoroughly homogenized by manual or mechanical agitation. The sulfuric acid is phased out and the clean extract removed for further cleanup techniques or analysis.

3.2 All PCB hexane extracts including blanks, matrix spikes and laboratory-fortified blanks must be subjected to the same cleanup.

4.0 PARAMETER OR COMPOUND LIST

4.1 PCB Aroclors include the following:

Compound	CAS Registry No.
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-03-128, *Semi-Volatile Laboratory Gas Chromatography Analysis of Polychlorinated Biphenyls (PCB)*, latest revision

5.2 TriMatrix SOP GR-09-109, *Sulfur Cleanup*, latest revision

5.3 TriMatrix SOP GR-09-120, *Florisil/Silica Gel Column Cleanup of PCBs, Toxaphene and Chlordane*, latest revision

5.4 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal,* latest revision

5.5 TriMatrix SOP GR-10-125, *Method Detection Limit (MDS)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Sulfuric acid cleanup will not remove chlorinated benzenes, chlorinated naphthalene (halowaxes), and some chlorinated pesticides.

7.0 SAFETY PRECAUTIONS

7.1 Wear a laboratory coat and approved safety glasses at all times in the organic extractions laboratory. In addition, wear disposable gloves when samples, extracts or reagents are handled.

7.2 Follow all safety instructions as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.3 Use extreme caution when handling concentrated sulfuric acid. The chemical can cause severe burns and destroy clothing. If exposed to sulfuric acid or any other chemical, flush with water for at least 15 minutes.

7.4 Clean up chemical spills immediately.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 There is no sample collection, preservation or handling directly associated with this procedure.

9.0 INSTRUMENTATION, APPARATUS, AND MATERIALS

9.1 Disposable glass Pasteur pipettes, 1 mL

9.2 Autosampler vials with PTFE-lined silicon septa, screw-cap lids, 2 mL

9.3 Vortex mixer

9.4 Micro-syringe, gas-tight, 1000 μL

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 There is no preventive maintenance directly associated with this procedure.

11.0 CHEMICALS AND REAGENTS

11.1 Concentrated sulfuric acid, ACS/reagent grade or better

11.2 Hexane, pesticides grade or better

12.0 STANDARDS PREPARATION

12.1 There is no standards preparation directly associated with this procedure.

13.0 ANALYTICAL PROCEDURE

13.1 Add 100-500 µL of concentrated sulfuric acid to a 2 mL vial and then add 0.5-1.0 mL of extract. Cap the vial tightly and vortex for 30-60 seconds on the vortex mixer. A swirling single-phase vortex will be visible in the vial for proper homogenization.

CAUTION: Stop vortexing immediately if a vial leaks from gas generation or exothermic heat is produced. Carefully and slowly open the vial to vent pressure before continuing the homogenization. **AVOID SKIN CONTACT WITH SULFURIC ACID**

13.2 After vortexing, let the vial sit to separate phases for at least one minute. Examine the top (hexane) phase. It should be clear. It should not be darkly colored, or have visible emulsion or cloudiness.

13.3 If a clear hexane extract is visible, let the phases separate completely and transfer to a clean 2 mL autosampler vial. Seal with the PTFE-lined cap.

13.4 If an extract remains darkly colored or an emulsion persists for several minutes, transfer to a clean 2 mL vial containing 100-500 µL of concentrated sulfuric acid and repeat the cleanup.

13.4.1 Repeat until the extract is clear or to a maximum of three times.

13.4.2 If an extract remains darkly colored after the third time, perform Florisil/Silica Gel cleanup by TriMatrix SOP GR-09-120, on a fresh extract aliquot that has been acid cleaned once.

13.4.3 When complete, carefully remove the hexane by letting the phases separate before pipetting out.

13.4.4 Be sure to leave all acid behind by not removing all the hexane layer.

13.5 After sulfuric acid cleanup, remove sulfur in accordance with TriMatrix SOP GR-09-109.

13.6 When cleanup is complete, record the sample number with all associated QC in the cleanup logbook.

13.7 Place cleaned extracts in the GC laboratory refrigerator to store at 4 ±2° C.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Record all cleanups in the organic extractions laboratory cleanup logbook.

15.0 QUALITY ASSURANCE

15.1 PCB extracts cleaned up by this procedure must have acceptable surrogate recovery. If surrogate recovery fails due to extract cleanup, correct the technique and repeat, or narrate results appropriately.

15.2 All PCB quality control must undergo the sulfuric acid cleanup, including blank spikes, matrix spikes and extraction blanks.

15.3 Match quality control cleanup with the number of times sample cleanup is performed.

15.4 Quality control recovery and contamination for extraction/cleanup is monitored at analysis.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 An Initial Demonstration of Capability (IDC) Study is required for each analyst to demonstrate the ability to generate acceptable accuracy and precision.

16.1.1 Prepare a PCB 1016/1260 spiking solution at a concentration that will put extract concentration in the middle of the instrument calibration range. Do not use the same standards used for calibration.

16.1.2 Spike four, 1 L aliquots of laboratory reagent water and extract as samples.

16.1.3 Clean up the four extracts following every step in this procedure.

16.1.4 After analysis of the four spikes, input results to the IDC spreadsheet located on the laboratory intranet library to calculate average percent recovery and relative standard deviation.

16.1.5 Average percent recovery must fall within blank spike acceptance limits and relative standard deviation must be ≤20%.

16.1.6 If either criterion fails, locate and correct the problem then repeat the study successfully.

16.1.7 Repeated failure however, will confirm a general problem with the procedure and/or techniques used. If repeated failure occurs, correct the procedure and/or techniques used then repeat the study successfully.

16.1.8 Analysts may not clean up extracts until an IDC has been successfully completed.

16.1.9 Submit a copy of successful IDC spreadsheets and raw data to Quality Assurance for training documentation.

16.2 A Continuing Demonstration of Capability (CDC) Study must be completed annually based on any of the following approaches:

16.2.1 By repeating the IDC study.

16.2.2 By using the last four results from an MDL study if run exclusively by the analyst.

16.2.3 By using four consecutive blank spike results obtained from routine sample cleanup if run exclusively by the analyst and from a source different than the instrument calibration.

16.2.4 By the successful completion of a Performance Testing sample if run exclusively by the analyst.

16.2.5 Use the IDC spreadsheet to calculate results when appropriate. Submit a copy of successful IDC spreadsheets and raw data to Quality Assurance for training documentation.

16.3 All Method Detection Limit (MDL) Studies must include the full cleanup of an extract in accordance with TriMatrix SOP GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory to monitor chemicals in the laboratory.

17.2 Never dispose of a chemical without first referencing appropriate disposal instructions for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of any chemical. An MSDS library is maintained on the laboratory intranet.

18.2 To minimize environmental impact and cost associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instruction in TriMatrix Laboratory SOP GR-15-102, for laboratory waste disposal.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 1, December, 1996, Method 3665A, "Sulfuric Acid/Permanganate Cleanup"

20.0 ATTACHMENTS

20.1 PCB Cleanup Logbook Example

Attachment 20.1
PCB Cleanup Logbook Example

TriMatrix Laboratories, Inc.

PCB Cleanup Logbook

Date	Analyst	Client	Sample Numbers	H_2SO_4	Copper	Combined Cleanup Using 1g Florisil/ 1g Silica Gel/Sodium Sulfate Plug: Florisil	Silica Gel	Sodium Sulfate	Other (Large Florisil, Cartridge, etc.) Include Reagent Numbers and g Used
11.7.08	DCG	[redacted]	0811004-01	8020963	8040629				
↓	↓	[redacted]	BLK, BS 0811034-01	↓	↓				
		[redacted]	MS, MSD 0811039-01						
		[redacted]	0810646-02 BLK 0810631-01 BS			PL 21.31-12	9020234		
11/11/08	BTH	[redacted]	0811183-01-02, MS, MSD	↓	↓				
11-12-08	DCG	[redacted]	BLK, BS, MS, MSD 0811094-01 TO -14	8020963	8040629				
↓	↓	[redacted]	0811099-01	↓	↓				
		[redacted]	11-12 BLK BS 0811105-04, -11						
		[redacted]	0811106-02						
11/14/08	BTH	[redacted]	0811286-01-19, 31, BLK, BS, MS, MSD	8020963	8040629				
↓	DCG	↓	BLK BS 0811286-34 TO -37	↓	↓				
	DCG		BLK BS MS MSD MSD2 32:33 0811281-20 TO 30						
11/26/08	BTH	[redacted]	0811279-01, BLK, BS	↓	↓	PL 21.31-12	8020234		
↓	↓	[redacted]	0811298-20			↓	↓		
12/1/08	ABC	[redacted]	BLK, BS, 0812001-01-06	↓	↓				
12/2/08	BTH	[redacted]	0812012-01-02, BLK BS	↓	↓				

file: pcblogbook1 page 15 of 25 revision 1.2

Approved By: TDJ 12-15-08
QA Officer

Approved By: BTH 12/15/08
Area Supervisor

TriMatrix Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Extraction of Organochlorine Pesticides, PCBs and Chlorinated Hydrocarbons from Water

SW-846 Method 3510C
EPA Method 608
EPA Method 612

APPROVALS:

Area Supervisor: Brian J. Hall — Date: 8/10/09

QA Officer: Tom C. Boocher — Date: 8-10-09

Operations Manager: Jeff P. Glaser — Date: 8/10/09

Procedure Number: GR-09-107
Revision Number: 5.2

Date Initiated: 3/30/94
Effective Date: 8/12/09

Date Revised: 7/27/09
Pages Revised: All

By: Andrea S. Colborn
Total Number of Pages: 22

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure has detailed instructions for extracting organochlorine pesticides and polychlorinated biphenyls and chlorinated hydrocarbons from aqueous samples using methylene chloride as the extraction solvent. The procedure also describes the concentration technique suitable for preparing extracts for analysis.

1.2 This procedure is restricted to use by or under the supervision of a trained analyst. Each analyst must demonstrate the ability to generate acceptable results completion of a demonstration of capability study before actual sample extractions may be performed.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Method 3510C, Revision 3, December 1996, "Separatory Funnel Liquid-Liquid Extraction"

2.2 *40 Code of Federal Regulations*, latest edition, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 608, "Organochlorine Pesticides and PCBs"

2.3 *40 Code of Federal Regulations*, latest edition, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 612, "Chlorinated Hydrocarbons"

3.0 SUMMARY OF PROCEDURE

3.1 A measured volume of sample, usually one liter, is serially extracted at neutral pH with methylene chloride using a 2 L glass separatory funnel.

3.2 Extracts are dried with sodium sulfate and concentrated with Kuderna-Danish concentration glassware.

3.3 Solvent exchange to hexane is performed and the extract is again concentrated to a final volume of 2.0 mL.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to TriMatrix SOP GR-03-120 and SOP GR-03-128 for parameters applicable to this procedure.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-03-128, *Gas Chromatography Analysis of Polychlorinated Biphenyls (PCBs)*, latest revision

5.2 TriMatrix SOP GR-03-120, *Gas Chromatography Analysis of Organochlorine Pesticides*, latest revision

5.3 TriMatrix SOP GR-09-109, *Sulfur Cleanup*, latest revision

5.4 TriMatrix SOP number GR-09-110, *Sulfuric Acid Cleanup*, latest revision

5.5 TriMatrix SOP GR-09-120, *Florisil/Silica Gel Column Cleanup of PCB's, Toxaphene, and Chlordane*, latest revision

5.6 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration,* latest revision

5.7 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.8 TriMatrix SOP GR-16-100, *Glassware Cleaning and Preparation for the Organics Extraction Laboratory*, latest revision

5.9 TriMatrix SOP GR-09-111, *Florisil Column Cleanup,* latest revision

5.10 TriMatrix SOP GR-04-101, *semi-Volatile Organic Laboratory Corrective Actions*, latest revision

5.11 TriMatrix SOP GR-10-125, *Method Detection Limit (MDL)*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interferences can be caused by contaminants in solvents, reagents, glassware or sample processing hardware leading to discrete artifacts or elevated baselines. All materials must routinely demonstrate freedom from interferences by analysis of laboratory reagent blanks.

6.1.1 Glassware must be scrupulously cleaned in accordance with TriMatrix SOP GR-16-100.

6.1.2 Use only high purity reagents and pesticides-grade solvents to minimize interferences.

6.2 Matrix interference may be caused by contaminants co-extracted from samples. Matrix interference will vary considerably from matrix to matrix. Such interference can affect analyte recovery and re-extraction or other corrective action may be necessary if surrogates and/or other spiked compounds fail established laboratory recovery limits.

6.3 Phthalate esters cause a direct interference with analyte extractions. Avoid using flexible plastics in contact with solvent to minimize this type of interference.

7.0 SAFETY PRECAUTIONS

7.1 Wear a laboratory coat and approved safety glasses while in the organic extractions laboratory. In addition, disposable gloves must be worn whenever samples or reagents are handled.

7.2 Follow all instructions outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.3 For laboratory waste disposal, refer to TriMatrix SOP GR-15-102.

7.4 The total toxicity and/or carcinogenicity of reagents used in this procedure have not been precisely defined.

7.4.1 Treat all chemicals as a potential health hazard.

7.4.2 Reduce expose to the lowest possible level by adherence to established safety policies.

7.4.3 Material Safety Data Sheets are maintained on the laboratory intranet of all chemicals used in this procedure. Consult the MSDS for detailed chemical information.

7.5 Samples can be highly toxic and varied. Treat any exposure as a potential danger and immediately decontaminate the exposure. Clean contaminated personal protective equipment before using again.

7.6 Bring all safety issues to the attention of the Area Supervisor and/or Health and Safety Officer.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples are collected in 1000 mL screw-cap amber glass jars with PTFE-lined lids. No preservative is required.

8.2 All samples must be extracted within seven days of the collection date, and analyzed within 40 days of extraction except for method 608 PCB wastewater samples which have a year to extract.

8.3 When not in use, samples must be stored in the walk-in cooler, at 4 ±2° C.

8.4 Analysts must use care when handling sample containers to avoid loss due to breakage.

8.5 After extraction and/or cleanup, samples must be stored in the GC laboratory refrigerator at 4 ±2° C until analysis.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Erlenmeyer flasks: 300 mL

9.2 Separatory funnels: 2 L glass with PTFE stopcock and PTFE stopper

9.3 Filtering funnels: 100 mm

9.4 Filter paper: Fisher P8, coarse or equivalent

9.5 Kuderna-Danish (K-D) concentrator glassware

9.5.1 Concentrator tubes: 10 mL, graduated (Kontes K-570050-1025 or equivalent)

9.5.2 Concentrator flasks: 500 mL (Kontes K-570001-500 or equivalent)

9.5.3 Concentrator tube clips

9.5.4 Snyder columns: Three-ball macro (Kontes K-50300-0121 or equivalent)

9.5.5 Snyder columns: Two-ball micro

9.6 Auto-sampler vials: 4.0 mL with PTFE-lined screw cap lids, pre-calibrated to 2.0 mL as outlined in TriMatrix SOP GR-09-106.

9.7 Boiling chips: PTFE, methylene chloride rinsed

9.8 pH paper: wide-range (0-14)

9.9 Phase separation paper

9.10 N-EVAP nitrogen evaporation bath

9.11 Pasteur pipettes: 2 mL disposable, glass

9.12 Water bath: variable temperature, capable of maintaining temperature to ±5° C

9.13 Syringes: 100 μL, 500 μL, 1000 μL

9.14 Graduated cylinders: 1 L

9.15 Analytical Balance, capable of accurately weighing 0.0001 g

9.16 Hot Plate: capable of variable temperature control to ±5° C

9.17 Glass wool

9.18 Volumetric flasks: 10 mL, 50 mL, 100 mL

9.19 Vials: 40 mL amber, with PTFE-lined screw cap lids

9.20 Centrifuge: variable speed

9.21 Centrifuge tubes: 40 mL, PTFE

9.22 Sodium sulfate rinsing equipment:

9.22.1 Buchner funnel: 20 cm

9.22.2 Flask: 4 L Vacuum

9.22.3 Drying pan: 13 x 9 inch, metal

9.22.4 Vacuum pump

9.22.5 Drying oven

9.22.6 Stopper: PTFE with hole for the Buchner funnel

9.22.7 Clamps: supporting

9.22.8 Filter paper: qualitative fast, 20 cm

9.22.9 Squirt bottle: PTFE

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 There is no preventive maintenance directly associated with this procedure.

11.0 CHEMICALS AND REAGENTS

11.1 Laboratory reagent water

11.1.1 Reagent water is laboratory-purified and methylene chloride extracted (unless otherwise specified) water in which no interferences are present at or above any reporting limit.

11.1.2 All reagent water used to dilute samples and blanks must be pre-extracted with methylene chloride to minimize interference from phthalate ester contamination:

11.1.2.1 Add 1000 mL of laboratory grade water to a 1000 mL separatory funnel

11.1.2.2 Add 60 mL of methylene chloride and extract by vigorously shaking with frequent venting.

11.1.2.3 Let the methylene chloride settle for at least 10 minutes then drain and discard.

11.1.2.4 The remaining mixture is then ready for diluting samples and for use as blanks.

11.2 Methylene chloride: Pesticide grade or equivalent

11.3 Hexane: Pesticide grade or equivalent

11.4 Sodium sulfate: ASC grade, anhydrous granular, rinsed:

11.4.1 This reagent must be rinsed before use. Assemble the sodium sulfate rinsing apparatus and clamp securely.

11.4.2 Insert filter paper in the Buchner funnel and add the entire contents of a 2.5 kg sodium sulfate container to the funnel.

11.4.3 Add 1 L of methylene chloride to the glass sodium sulfate container, rinsing down the inside with methylene chloride from a PTFE squirt bottle.

11.4.4 Pour this methylene chloride over the sodium sulfate in the Buchner funnel, letting it drain without applying vacuum. Add more methylene chloride if necessary to completely immerse the sodium sulfate.

11.4.5 After most of the solvent has drained, apply vacuum and rinse with an additional 100 mL from the PTFE squirt bottle. Maintain vacuum until solvent stops draining.

11.4.6 Transfer the rinsed sodium sulfate to a drying pan and heat in a drying oven at 120° C for at least one hour.

11.4.7 Remove from the oven with heat-resistant gloves and cool in a hood. Always place a caution sign by the pan while cooling.

11.4.8 After cooling, return to the original container using a glass funnel.

11.4.9 Label the container "Rinsed" with the organic preparation laboratory reagent identification (ID) number then place it in reagent storage.

11.5 Sodium Hydroxide solution (10N): Carefully, dissolve 400 g sodium hydroxide pellets in 1 L of reagent water.

CAUTION: This solution generates significant heat. Add pellets slowly and cool the solution before using.

11.6 Sulfuric acid solution (1:1 v/v): Carefully, add 500 mL concentrated sulfuric acid to 500 mL reagent water in a glass container.

CAUTION: This solution generates significant heat. Add acid slowly to the water and cool before using. NEVER add water to acid.

12.0 STANDARDS PREPARATION

12.1 Laboratory-made surrogates and spike solutions are made by the following rules:

12.1.1 Obtain glassware and materials required for surrogates or spike solutions being made.

12.1.2 Use the appropriate solvent for dilution.

12.1.3 Label all glassware and vials holding surrogate/spiking solutions.

12.1.4 Use minimal headspace in vials.

12.2 Surrogate and spiking solutions are stored in 40 mL narrow-mouth amber vials, labeled with the following information:

12.2.1 Surrogate or spike name

12.2.2 Laboratory-assigned surrogate or spike ID

12.2.3 Date made

12.2.4 Analyst initials

12.2.5 Solvent

12.2.6 Concentration and units

12.2.7 Expiration date

12.3 Using a microsyringe, inject the stock standard into a volumetric flask approximately half full of the appropriate solvent. For all standards, use only pesticide grade or better solvents. Make sure the syringe tip is below the solvent surface when injecting. Invert enough times to mix thoroughly. All surrogate and spiking standards are stored in 40 mL amber jars with PTFE-lined lids.

12.4 Surrogate/spike standards data is recorded in the extraction standards log. Surrogate/spiking solutions are stored in the organic extractions laboratory refrigerator at 4 ±2°C. A dilution of the spiking solution must be analyzed by the analytical laboratory to verify the concentration prior to use. The concentration recovered must be within 80 – 120%.

12.5 Neat compounds must be ACS grade or better and be weighed with the calibrated analytical balance in the hood. If purity of a neat standard is below 95%, adjust the concentration in all subsequent calculations. Do not use any neat compound past its expiration date. If using the last of a compound, see that it is promptly re-ordered before it runs out.

12.5.1 For neat solids, weigh to the nearest 0.0001 g in a volumetric flask and record the mass. Swirl the flask to mix after adding solvent. If a compound does not dissolve, place the flask in a small sonicator bath to aid in dissolution. Closely monitor the solution and remove as soon as the compound has dissolved. Do not over-sonicate as excessive heat will be generated. Only after complete dissolution, dilute to volume and mix well by inverting at least three times. Discard the volume in the flask neck and transfer the rest to pre-labeled vials with minimal headspace. Store the vials at 4 ±2° C.

12.5.2 For neat liquids, weigh into a tared volumetric flask half-full of the appropriate solvent. Add the neat liquid drop-wise using a Pasteur pipette directly into the solvent, being careful not to touch the inside of the flask neck. Record the mass, swirl to mix then dilute to volume. Mix well and discard the volume in the flask neck. Transfer the rest to pre-labeled vials with minimal headspace and store at 4 ±2° C.

12.6 The shelf-life of surrogate and spiking solutions is six months for working solutions and one year for stock concentrations. Dispose of any solution sooner if a manufacturer's expiration date occurs within that time for any neat material used. Once a chemical or solution has expired, it must be removed from the laboratory and disposed of. Monitor the expiration date of all chemicals and solutions. To avoid costly rush shipping, it is important to promptly order more chemical once consumed.

12.7 Often when making surrogate and spiking solutions, serial dilutions are required to achieve proper concentration. Serial dilutions are made from a higher concentration. The following example illustrates making a 1.0 mg/L working solution from a 10,000 mg/L stock concentration:

$$10000 \text{ mg/L} \times \frac{1 \text{ mL stock standard}}{100 \text{ mL final volume}} = 100 \text{ mg/L}$$

$$100 \text{ mg/L} \times \frac{1 \text{ mL stock standard}}{100 \text{ mL final volume}} = 1.0 \text{ mg/L}$$

12.8 When making a dilution, all data must be entered into the standards log in Element™ which is the laboratory information management system (LIMS) .

12.8.1 Refer to Attachment 20.2 for an example standards log entry.

12.8.2 Record each serial dilution made from a solution with a full explanation of what was done.

12.8.3 If a dilution of a dilution is made then the resultant solution would be given another Element™ number. This nomenclature is continued for each subsequent dilution.

12.8.4 For solution preparations, use only pesticide-grade or better solvents.

12.8.5 Before using a solution, the calculation performed when calculating concentrations must be verified by a second analyst. Once the calculation has been verified, solution concentration must be verified by actual analysis. If analytical recovery is not within 80 – 120%, the solution may not be used.

12.9 Surrogate solution concentration:

12.9.1 Surrogates are made from purchased solutions containing both surrogates and diluted to 0.20 mg/L. Water samples are spiked with 1.0 mL of the dilution to give extracts the same concentration since final extract volume is 2.0 mL.

Compound(s)	Source	Solvent	Stock Concentration	Stock Dilution	Working Concentration
2,4,5,6-Tetrachloro-m-xylene and Decachlorobiphenyl	Absolute Standards (#20023)	Acetone	200 mg/L	100 μL:100 mL	0.20 mg/L

12.10 Pesticide analysis spike solution concentration:

12.10.1 Spikes are made from purchased solutions and diluted to 0.20 mg/L. Water samples are spiked with 1.0 mL of the dilution to give extracts the same concentration as surrogates since final extract volume is 2.0 mL.

Compound(s)	Source	Solvent	Stock Conc.	Stock Dilution	Working Conc.
TCL Pesticides	Purchased	Methanol	1000 mg/L	80 μL:100 mL	0.80 mg/L

12.11 PCB analysis spike solution concentration:

12.11.1 Spikes are made from purchased solutions and diluted to 1.0 mg/L. Water samples are spiked with 1.0 mL of the dilution to give extracts a concentration of 0.50 mg/L.

Compound(s)	Source	Solvent	Stock Conc.	Stock Dilution	Working Conc.
PCBs 1016, 1221, 1232, 1242 1248, 1254, 1260	NSI	Acetone	1000 mg/L	1:1000	1.0 mg/L

12.12 Chlorinated Hydrocarbon Spike Solution is made from purchased solution at vary concentrations:

Compound(s)	Source	Stock Conc.	Stock Dilution	Working Conc.
2-Chloronaphthalene	O_2SI	2500	20 μL:25 mL	2 mg/L
1,2,3-Trichlorobenzene	O_2SI	100	21 μL:25 mL	0.08 mg/L
1,2,4,5-Tetrachlorobenzene	O_2SI	100	22 μL:25 mL	0.08 mg/L
1,2,4-Trichlorobenzene	O_2SI	100	23 μL:25 mL	0.08 mg/L
1,2-Dichlorobenzene	O_2SI	2500	24 μL:25 mL	2 mg/L
1,3,5-Trichlorobenzene	O_2SI	100	25 μL:25 mL	0.08 mg/L
1,2,3,4-Tetrachlorobenzene	O_2SI	100	26 μL:25 mL	0.08 mg/L
1,4-Dichlorobenzene	O_2SI	2500	27 μL:25 mL	2 mg/L
Pentachloronitrobenzene	O_2SI	100	28 μL:25 mL	0.08 mg/L
Hexabromobenzene	O_2SI	100	29 μL:25 mL	0.08 mg/L
Hexachlorobenzene	O_2SI	100	30 μL:25 mL	0.08 mg/L
Hexachlorobutadiene	O_2SI	100	31 μL:25 mL	0.08 mg/L
Hexachlorocyclopentadiene	O_2SI	100	32 μL:25 mL	0.08 mg/L
Hexachloroethane	O_2SI	100	33 μL:25 mL	0.08 mg/L
Pentachlorobenzene	O_2SI	100	34 μL:25 mL	0.08 mg/L
1,3-Dichlorobenzene	O_2SI	2500	35 μL:25 mL	2 mg/L

13.0 ANALYTICAL PROCEDURE

13.1 Assemble the glassware and samples.

13.1.1 Rinse all glassware with methylene chloride prior to use.

13.1.2 Label each separatory funnel, Erlenmeyer flask, K-D concentrator and concentrator tube with the following:

13.1.2.1 The sample ID number

13.1.2.2 The analysis

13.1.2.3 "S" for surrogate addition (Erlenmeyer flasks only)

13.1.2.4 "L" for spike addition, where applicable (Erlenmeyer flasks only)

13.1.2.5 The final extract volume and units

13.2 Determine the sample volume as follows:

13.2.1 Shake all sample jars well. If a shaken sample remains clear, mark the water level on the outside of the jar then transfer its entire contents to a separatory funnel. Do not discard the sample jar. After extraction is complete, fill the jar with tap water to the water level mark (made before the sample was emptied) then measure with a graduated cylinder to attain the sample volume.

13.2.2 If a shaken sample is so cloudy it is difficult to see through the jar, let the sediment settle for approximately five minutes then decant the aqueous portion into a clean graduated cylinder to measure the volume. Shake any sediment remaining in the jar into the trash. DO NOT RINSE A SAMPLE JAR TO REMOVE SEDIMENT OR RESIDUE. After the sample volume has been measured and recorded, transfer the graduated cylinder contents to a separatory funnel.

Note: Any sample sediment suspected of containing PCBs and/or high concentrations of pesticides must be disposed of as hazardous waste in accordance with TriMatrix SOP GR-15-102.

13.2.3 If less than 500 mL of a sample is measured, consult the project chemist before extracting since less than 500 mL will result in elevated reporting limits, which must be pre-approved by the client.

Note: TCLP leachates extracted using 500 mL do not require consultation with the project chemist before extracting.

13.3 Extraction must be done at a pH of 5-9. Check the sample pH using wide-range pH paper by transferring a couple drops of sample onto the pH strip using a Pasteur pipette. Adjust the pH as necessary using sodium hydroxide or sulfuric acid solutions.

13.4 Add 1.0 mL of surrogate solution to each separatory funnel with a 1.0 mL microsyringe.

13.4.1 Surrogate is added to all extractions.

13.4.2 Do not submerge the microsyringe needle into the liquid. Slowly inject approximately one inch above the liquid's surface.

13.4.3 Do not inject too fast since splashing may occur. If splashing occurs onto the syringe, rinse it internally and externally with methanol. Discard the rinsate.

13.4.4 Cross out the "S" on each corresponding Erlenmeyer as surrogate is added.

13.5 Add 1.0 mL PCB spiking solution to matrix spikes, matrix spike duplicates and blank spikes if the extraction batch is for PCB analysis. Cross out the "L" on each corresponding Erlenmeyer as spike is added.

13.6 Add 100 μL pesticide spiking solution to matrix spikes, matrix spike duplicates and blank spikes if the extraction batch is for pesticides analysis. Cross out the "L" on each corresponding Erlenmeyer as spike is added.

13.7 Add 1.0 mL chlorinated hydrocarbons spiking solution to matrix spikes, matrix spike duplicates and blank spikes if the extraction batch is for chlorinated hydrocarbons analysis. Cross out the "L" on each corresponding Erlenmeyer as spike is added.

13.8 Rinse sample jars and graduated cylinders (when cylinders are used for individual samples) with methylene chloride as follows:

13.8.1 Add 100 mL methylene chloride to each sample jar. Replace the lid (very important).

13.8.2 Shake and vent as necessary.

13.8.3 Pour into the corresponding graduated cylinders and swirl to rinse the sides.

13.8.4 Finally, pour from the graduated cylinder into the separatory funnel containing sample.

13.9 Extract the quality control and samples as follows.

13.9.1 Stopper each separatory funnel and invert. Immediately open stopcock to release pressure.

13.9.2 Close the stopcock, shake and vent again.

13.9.3 Repeat until there is no evidence of pressure.

13.9.4 Shake <u>vigorously</u> for an additional two minutes.

13.9.5 Let the separatory funnel sit for at least ten minutes to allow phase separation.

13.9.6 If an emulsion forms during extraction:

13.9.6.1 If the emulsion is minor drain the emulsion through phase separation paper into a 300 μL Erlenmeyer flask. When the phase separation paper is dripping slowly, rinse it with 20-30 mL of methylene chloride.

13.9.6.2 If separation does not occur and the emulsion looks like pudding, drain the entire contents of the emulsion into PTFE centrifuge tubes. Centrifuge for four minutes at 4500 rpm. Once centrifuged, transfer the water phase with any intermediary emulsion back into the 2 L funnel. Pour the bottom layer into a 300 mL Erlenmeyer flask.

13.9.7 If emulsion is minimal, drain the methylene chloride (bottom layer) directly into a 300 mL Erlenmeyer flask.

13.10 Repeat the extraction twice more with 60 mL aliquots of methylene chloride. Rinse the sample jars once more with the first of these solvent volumes.

13.11 Transfer the total extract volume to a K-D concentrator as follows:

13.11.1 Quantitatively transfer each extract from the 300 mL flasks through a funnel containing about one inch of sodium sulfate in P8 filter paper into a properly labeled Kuderna-Danish (K-D) concentrators with an attached 10 mL graduated concentrator tube.

13.11.2 Rinse the flasks with several 20-30 mL aliquots of methylene chloride to complete the transfer.

13.12 Concentrate the extracts as follows:

13.12.1 Add one or two clean boiling chips to each concentrator tube and attach a three-ball Snyder column.

13.12.2 Pre-wet the Snyder columns by adding approximately 1 mL of methylene chloride through the top.

13.12.3 Place the concentrators in a water bath (80-90° C) where the concentrator tube is partially immersed and the entire lower rounded surface of the K-D flask is bathed with vapor.

13.12.4 At the proper rate of distillation, balls of each column will actively chatter but not flood. Until chattering begins, rattle Snyder columns periodically.

13.13 Solvent exchange to hexane:

13.13.1 When the apparent volume of liquid reaches 3-5 mL (in ten to fifteen minutes), remove the Snyder column and quickly add 40-60 mL of hexane and two new boiling chips to each concentrator and replace the Snyder column. Add about 10 mL more hexane to the top of the Snyder column. Increase the temperature of the bath to 90-95° C. Concentrate again to an apparent volume of 5 mL. Remove and cool for at least 10 minutes. The volume in the concentrator tube after cooling should be approximately 8-10 mL and appropriate for micro-concentration.

13.14 Micro-Snyder column technique is used to adjust the final extract volume to 2.0 mL. However, if using nitrogen blowdown instead of the micro-Snyder column, proceed to Section 13.4.2.

13.14.1 Micro-Snyder Technique:

13.14.1.1 Remove each concentrator tube and rinse the ground-glass joint with hexane.

13.14.1.2 Add another clean boiling chip then attach a 2-ball micro-Snyder column.

13.14.1.3 Pre-wet the column with 0.5 mL hexane.

13.14.1.4 Place concentrator tubes in a water bath, partially immersed. The water temperature should be 90-95° F. Care should be taken to avoid bumping or column flooding due to immersing too deep.

13.14.1.5 When the apparent volume reaches 1 mL, remove from the bath. Cool for at least ten minutes.

13.14.1.6 Rinse the ground-glass joints with 0.2 mL hexane after removing the Snyder column.

13.14.1.7 Transfer each extract to a 2.0 mL calibrated vial using a clean, disposable, Pasteur pipette.

13.14.1.8 Using approximately 0.8 mL of hexane, rinse each concentrator tube and transfer to the vial.

13.14.1.9 Adjust the final volume to 2.0 mL then cap tightly.

13.14.1.10 Extracts are ready for cleanup or analysis. Store extracts to be cleaned up in the refrigerator.

13.14.2 If using nitrogen blowdown instead of the micro-Snyder column, proceeed as follows:

13.14.2.1 Position sample bottles on the N-EVAP.

13.14.2.2 With a gentle stream of nitrogen, concentrate extracts to <u>not less than</u> 2.0 mL.

13.14.2.3 Extracts are ready for cleanup or analysis. Store extracts to be cleaned up in the refrigerator.

13.15 Extract cleanup

13.15.1 If necessary, pesticide extracts may be cleaned up using Florisil columns (1000 mg or 20 g) in compliance with TriMatrix SOP GR-09-111.

13.15.2 All PCB extracts must be cleaned up as follows:

13.15.2.1 Sulfuric acid cleanup in compliance with TriMatrix SOP GR-09-110.

13.15.2.2 Copper powder to remove elemental sulfur, sulfur cleanup in compliance with TriMatrix SOP GR-09-109.

13.15.3 If PCB extracts have color and/or do not appear to be free of contaminants after sulfuric acid and copper, then the extracts need cleaned up as follows and in the order listed:

13.15.3.1 Florisil/Silica gel column cleanup in compliance with TriMatrix SOP GR-09-120.

13.15.3.2 Sulfuric acid cleanup in compliance with TriMatrix SOP GR-09-110.

13.15.3.3 Sulfur cleanup in compliance with TriMatrix SOP GR-09-109.

13.16 Store cleaned up extracts at 4 ±2° C in the GC refrigerator.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Extraction analysts are responsible for extraction documentation and data integrity. All documentation must be correctly filled in. It is important to document extractions by correctly filling in, turning in and filing accurate paperwork. This is mandatory for quality control and to provide clients with defensible data.

14.2 Report extractions as follows:

14.2.1 Input all extraction data to Element™. Extraction reports must be filled in completely to insure that results are reported correctly and data is associated with the right quality control batch number.

14.2.2 If internal chain-of-custody (CoC) is required, it is very important that the CoC form be filled in completely and correctly.

14.3 Complete all laboratory records as follows:

14.3.1 Logbooks must be filled in completely and correctly. Corrections are to be made with a line-out, not a write-over or scribble-out. Blank lines in the logbook must be Z'd out.

14.3.2 Transfer extraction summary benchsheets (including CoC forms) to the GC/MS analyst with finished extracts.

15.0 QUALITY ASSURANCE

15.1 An extraction blank (BLK) and blank spike (BS) must be done daily or for each extraction batch of up to 20 samples, whichever comes first, to demonstrate that extraction interferences are under control.

15.2 Matrix spikes (MS) and matrix spike duplicates (MSD) are extracted at a minimum of each extraction batch of up to 20 samples for analysis group or once a week, whichever is more frequent and provided enough sample is received. However, matrix spikes (MS) and matrix spike duplicates (MSD) are extracted at a minimum of each extraction batch of up to 10 samples for method 608 (primarily wastewater effluent samples) or once a week, whichever is more frequent and provided enough sample is received.

15.2.1 Performance records must be maintained to document the data quality generated.

15.2.2 Matrix spikes are extracted by measuring 1.0 mL of PCB spike solution (or 100 μL of pesticides spike solution) in addition to 1.0 mL of surrogate, to 1 L of sample (or 1.0 mL 8121 spiking solution).

15.3 Blank spikes (BS) are prepared by measuring 1.0 mL of PCB spike solution (or 100 μL of pesticides spike solution) in addition to 1.0 mL of surrogate, into 1 L of organic-free laboratory reagent water (or 1.0 mL 8121 spiking solution).

15.4 Matrix interference from samples or from laboratory-induced contamination may affect analyte recovery. Investigation in accordance with TriMatrix SOP GR-04-101 may be necessary if surrogate and/or spiked compounds fail laboratory-established control limits.

15.5 Surrogates must be added to all extractions.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before processing actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by successfully completing an Initial Demonstration of Capability (IDC) study. A Continuing Demonstration of Capability (CDC) is required annually.

16.1.1 Initial Demonstration of Capability

16.1.1.1 Prepare a spiking solution at analyte concentrations listed in Section 12.0. Prepare the spiking solution separately from the instrumental calibration standards. Spike 1.0 mL (100 μL for pesticides) of the solution into four, 1 L aliquots of water and extract following all steps in this procedure. After extracting, have the extracts analyzed then input the results to the IDC spreadsheet located on the laboratory intranet library. Average percent recovery as calculated from the spreadsheet must fall within the blank spike acceptability window listed in Element™. Standard deviation of the average recovery must be ≤20%.

16.1.1.2 If the study fails for either recovery or percent difference, locate and correct the source of the problem and repeat the study. If the second study passes, the analyst has demonstrated the procedure successfully. Repeated failure however, will indicate a procedure or technique problem. If this occurs, correct the technique or procedure and repeat the study successfully.

16.1.1.3 Samples may not be processed by any analyst or on any instrument until an IDC study has been successfully completed. Copies of successful demonstrations of capability spreadsheets and raw data must be provided to Quality Assurance (QA) for documentation of training.

16.1.2 Continuing Demonstration of Capability (CDC)

16.1.2.1 Annually, a CDC is required by each analyst. The CDC may be accomplished by repeating the IDC study, using the last four results from an exclusively run MDL study or by extracting a successful blind PT study sample. Input results to the same spreadsheet used for IDC studies.

16.2 A Method Detection Limit (MDL) study is required annually in accordance with TriMatrix SOP GR-10-125.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in TriMatrix SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update III, Method 3510C, Revision 3, December 1996, "Separatory Funnel Liquid-Liquid Extraction"

19.2 *40 Code of Federal Regulations*, most current edition, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 608, "Organochlorine Pesticides and PCBs"

19.3 *40 Code of Federal Regulations*, most current edition, Part 136, Appendix A, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Method 612, "Chlorinated Hydrocarbons"

20.0 ATTACHMENTS

20.1 Preparation Batch Report Example

20.2 Extraction Standards Log Entry Example

20.3 Organic Preparation Laboratory Waters Logbook Example

20.4 Addendum to the Procedure in Reference to Sample Batching

Attachment 20.1
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0703579 Page 1 of 1

Printed: 4/9/2007 4:54:47PM

Semivolatiles GC, Waste Water, 608 Liquid/Liquid Extraction

Surrogate #1 = 7010436 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0704032	608 PCBs (std 7 aroclors)	0704054	608 PCBs (master list)	0704064	608 PCBs ([redacted])
0704064	608 PCBs (std 7 aroclors)	0704064	608 PCBs (master list)	0704071	608 PCBs ([redacted])
0704081	608 PCBs (std 7 aroclors)				

Lab Number	Contain	Prepared	By	Initial (mL)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0703579-BLK1		Apr-06-07 13:00	BJH	1000	2	1000				BLANK	
0703579-BS1		Apr-06-07 13:00	BJH	1000	2	1000		7010798	1000	LCS	
0703579-MS1		Apr-06-07 13:00	BJH	1020	2	1000	0704064-02	7010798	1000	MATRIX SPIKE	
0703579-MSD1		Apr-06-07 13:00	BJH	1020	2	1000	0704064-02	7010798	1000	MATRIX SPIKE DUP	
0704032-01	A	Apr-06-07 13:00	BJH	1060	2	1000				[redacted]	report MPB at 0.1ug/L; plz add for TOT
0704054-02	B	Apr-06-07 13:00	BJH	1000	2	1000				[redacted]	[redacted]
0704064-01	C	Apr-06-07 13:00	BJH	1010	2	1000				[redacted]	
0704064-02	F	Apr-06-07 13:00	BJH	1020	2	1000				[redacted]	
0704064-02	F	Apr-06-07 13:00		1020	2	1000				[redacted]	Added for BatchQC in: 0703579
0704064-02	F	Apr-06-07 13:00		1020	2	1000				[redacted]	Added for BatchQC in: 0703579
0704064-04	E	Apr-06-07 13:00	BJH	1060	2	1000				[redacted]	
0704071-01	B	Apr-06-07 13:00	BJH	1020	2	1000				[redacted]	1254 only
0704081-01	C	Apr-06-07 13:00	BJH	1030	2	1000				[redacted]	
0704081-02	D	Apr-06-07 13:00	BJH	1030	2	1000				[redacted]	

Comments:	Analyst Initials:

bch_TriMatrix.rpt

Attachment 20.2
Extraction Standards Log Entry Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
7040565

Description:	1016/1260 Soil Spike	Expires:	Oct-16-07
Standard Type:	Analyte Spike	Prepared:	Apr-16-07
Solvent:	Hexane Lot #E08E21	Prepared By:	Brian J. Hall
Final Volume (mls)	100	Department:	Semivolatiles GC
Vials:	1	Last Edit:	Apr-16-07 15:21 by BJH

Analyte	CAS Number	Concentration	Units
PCB-1016	12674-11-2	10	ug/mL
PCB-1016 [2C]	12674-11-2	10	ug/mL
PCB-1260	11096-82-5	10	ug/mL
PCB-1260 [2C]	11096-82-5	10	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
A604465	(AMP) Aroclor 1016	May-12-06	** Vendor **	Sep-30-08	Apr-12-07 13:29 by JLW	1
A604466	(AMP) Aroclor 1260 stock	Oct-31-05	** Vendor **	Oct-31-08	Apr-12-07 13:35 by JLW	1

Reviewed By ______________________ Date

Page 1 of 1

Attachment 20.3
Organic Preparation Laboratory Waters Logbook Example

TriMatrix Laboratories, Inc.

Organic Extraction Lab
Water Extraction Logbook

Date: 4/9/07

Client	Project/ Submittal	Sample Number Range	LIMS Batch ID	Test or Parameter	Surrogate Spike Number and Volume		Matrix Spike Number and Volume	
[redacted]	0704064	BLK, BS, MS, MSD 0704064-01-04	0703613	3510/8270	6120791	100 ul	7030610	100 ul
[redacted]	0704095	0704095-05-07, 09	"	"	"	"		
[redacted]	0704100	0704100-01	"	"	"	"		
[redacted]	0703391	BLK, BS, 0703391-06RE	0703180	8151	A610087	100 ul	7030846	500 ul
ASC	4/9/07							

LIMS Sample ID	Bath Temp. °C	Initial Volume (mL)	Final Volume (mL)	Analyst Initials	LIMS Sample ID	Bath Temp. °C	Initial Volume (mL)	Final Volume (mL)	Analyst Initials
0703613BLK	70	1000	1.0	BJH					
0704064-01		1010							
0704064-02		1050							
0704064-03		1060							
0704064-04		1060							
0703613MS		1050							
0703613MSD		1050							
0703613BS		1000					ASC 4/9/07		
0704095-05	70	1060	1.0	BJH					
0704095-06		1010							
0704095-07		920							
0704095-09		970							
0704100-01	70	1060	1.0	BJH					
0703182BLK2	68	1000	5.0	ASC.					
0703391-06RE1		1030							
0703182BS2		1000							
		ASC 4/9/07							

Reagent Information

CH_2Cl_2 Lot #:	C40467	1:1 H_2SO_4 Reagent #:	PLR261-15
Hexane Lot #:	C50E54	1:1 HCl Reagent #:	NA
Na_2SO_4 Reagent #:	PLR23-2	10N NaOH Reagent #:	7040188
		NaCl Reagent #:	NA

file: Prep Lab Water Logbook page: 1 of 30 revision: 0.0

Approved By: [signature] 8-10-09 Approved By: BJH 8/10/09
QA Officer Area Supervisor

Attachment 20.4
Addendum to the Procedure in Reference to Sample Batching

Some clarification of method QC batching requirements and refinement of TriMatrix batching policies is necessary.

It is not permissible to extract more than 20 samples of the same method and matrix without the extraction of additional matrix QC samples.

To prevent this from happening, only one QC batch of a given method and matrix may be open at any one time. When reaching 20 samples or when a Level 3 project arrives, close the currently active batch and begin a new one. It does not matter if the current batch contains less than 20 samples. This policy will be applicable to all extracted samples with 2 exceptions:

#1) If multiple Level 3 and above clients come in on the same day, all of which have samples of the same matrix/method combination, it will be necessary to have more than one QC batch open. At the end of the day, close all the open batches except the one with the fewest samples. That one will remain open as the active batch.

#2) Certain clients send in samples over an extended time period. They also specify which of their samples are to be spiked. The selected sample does not always come in with the first round of samples received. For these clients and under these circumstances it will also be acceptable for multiple QC batches for the same method and matrix are open concurrently. Treat these batches as if they did not exist with regards to batching other samples.

Additionally, except for #2 above, batches may not be held open for longer than 7 days. After seven days close the active batch and begin a new one.

Approved By: MB 8-10-09 **Approved By:** BJH 8/10/09
QA Officer **Area Supervisor**

TriMatrix Laboratories, Inc.

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STANDARD OPERATING PROCEDURE

Extraction of Organochlorine Pesticides and Polychlorinated Biphenyls from Soil, Sludge and Wipe Samples

SW-846 Method 3550C

APPROVALS:

Area Supervisor: Brian J. Hall Date: 3/2/09

QA Officer: Tom C. Boocher Date: 2-27-09

Operations Manager: Jeff P. Glaser Date: 3/3/09

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If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to the extraction of chlorinated pesticides and polychlorinated biphenyls (PCB) from soil, sludge, and wipes.

1.2 Sonication is used to ensure sufficient contact with extraction solvent and facilitate acceptable analyte recovery from most sample matrices. Sonication is not appropriate where extraction efficiencies for low analyte concentrations are required.

1.3 If necessary and when appropriate, extracts will be cleaned up after extraction and concentration.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, February, 2007, Method 3550C, "Ultrasonic Extraction"

3.0 SUMMARY OF PROCEDURE

3.1 Extraction for analyte concentration at or below 20 mg/kg.

3.1.1 Anhydrous sodium sulfate is mixed with 30 g of sample to form a free-flowing solid.

3.1.2 Samples are serially extracted three times by sonication with 1:1 methylene chloride/acetone. Extracts are concentrated then solvent exchanged to hexane. Extracts are concentrated again to 10.0 mL for cleanup and/or GC analysis.

3.1.3 This technique is for low to medium analyte concentrations and negligible matrix interference. If necessary, final extract volumes may be greater than 10 mL or the following technique used.

3.2 Extraction procedure for analyte concentration above 20 mg/kg or difficult sample matrices

3.2.1 Samples with a history of high analyte concentration or matrix interference are extracted using 2.0 g of sample. Extraction analyst discretion based on experience is used to determine the need for this technique.

3.2.2 Anhydrous sodium sulfate is added to form a free-flowing solid.

3.2.3 Samples are extracted once by sonication with hexane. Extracts are filtered through glass wool and collected in a 15 mL vial for cleanup and/or gas chromatographic (GC) analysis.

3.3 Extraction procedure for wipes

3.3.1 Wipes are serially extracted three times by sonication with hexane in the sample container.

3.3.2 The hexane extract is concentrated to 10.0 mL for cleanup and/or GC analysis.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to TriMatrix SOP GR-03-120 for an organochlorine pesticide list associated with this procedure.

4.2 Refer to TriMatrix SOP GR-03-128 for a polychlorinated biphenyl (PCB) list associated with this procedure.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-03-128, *Semi-Volatile Laboratory Gas Chromatography Analysis of Polychlorinated Biphenyls (PCBs)*, latest revision

5.2 TriMatrix SOP GR-03-120, *Semi-Volatile Laboratory Gas Chromatography Analysis of Organochlorine Pesticides*, latest revision

5.3 TriMatrix SOP GR-09-109, *Sulfur Cleanup*, latest revision

5.4 TriMatrix SOP GR-09-110, *Sulfuric Acid Cleanup*, latest revision

5.5 TriMatrix SOP GR-09-111, *Florisil® Column Cleanup*, latest revision

5.6 TriMatrix SOP GR-09-120, *Florisil®/Silica Gel Column Cleanup of PCBs, Toxaphene and Chlordane*, latest revision

5.7 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.8 TriMatrix SOP GR-09-128, *Soil Mixing and Grinding*, latest revision

5.9 TriMatrix SOP GR-09-106, *Semi-Volatile Extract Vial Calibration*, latest revision

5.10 TriMatrix SOP GR-16-100, *Equipment Cleaning and Preparation for the Organic Extraction Laboratory*, latest revision

5.11 TriMatrix SOP GR-04-101, *Semi-Volatiles Laboratory Quality Control Corrective Actions*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 Interference can be caused by contaminants in solvents, reagents, glassware, or sample processing equipment, leading to discrete artifacts or elevated baselines. All materials used in this procedure must routinely demonstrate to be free from interference by analysis of blank spikes (BS).

6.2 Use only reagent-grade or better reagents, and pesticide-grade or better solvents. Clean all glassware in accordance with TriMatrix SOP GR-16-100.

6.3 Phthalate esters cause interference with pesticide and PCB analysis. Avoid using flexible plastics in contact with solvent to minimize this type of contamination.

6.4 Matrix interferences can affect analyte recovery. Repeating an extraction and/or cleanup may be necessary if surrogates or other spiked compounds fail laboratory established control limits.

7.0 SAFETY PRECAUTIONS

7.1 Extraction personnel must wear laboratory coats and approved safety glasses while in the organic extractions laboratory area. Also, disposable gloves must be worn whenever samples, reagents and/or solvents are handled.

7.2 Follow all instructions outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.3 For waste disposal refer to TriMatrix SOP GR-15-102.

7.4 The toxicity and/or carcinogenicity of chemicals used in this procedure has not been fully defined.

7.4.1 Treat all chemicals as a potential health hazard.

7.4.2 Reduce exposure to the lowest possible level by adherence to established safety practices.

7.4.3 A Material Safety Data Sheet (MSDS) is located on the laboratory intranet for all chemicals used in this procedure.

7.5 Bring safety issues to the attention of the Health and Safety Officer and Area Supervisor.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 Samples are collected in 60 mL, 125 mL or 250 mL wide-mouth glass jars with PTFE-lined screw-cap lids.

8.2 All samples must be extracted within fourteen days of the collection date and analyzed within 40 days of extraction.

8.3 When not in use, samples must be stored in the walk-in cooler at 4 ±2° C.

8.4 Use care when handling sample containers to avoid sample loss due to breakage.

8.5 After extraction and/or cleanup, store extracts in the GC refrigerator at 4 ±2° C until analysis.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Beakers, heavy-duty Pyrex: 400 mL and 600 mL

9.2 Ultrasonic disrupter:

9.2.1 Fisher model 550 with ¾ inch horn

9.2.2 For the high concentration extraction, use the 1/8 inch tapered microtip attached to a ½ inch horn

9.2.3 Sonabox, for hearing protection

9.3 Kuderna-Danish (K-D) concentrator glassware:

9.3.1 Concentrator tubes: 10 mL, graduated (Kontes K-570050-1025 or equivalent)

9.3.2 Concentrator flasks: 500 mL (Kontes K-570001-500 or equivalent)

9.3.3 Concentrator tube clips

9.3.4 Snyder columns: Three-ball macro (Kontes K-50300-0121 or equivalent)

9.3.5 Snyder columns: Two-ball micro

9.4 Water bath: Variable temperature

9.5 Balance, top-loading, capable of accurately weighing to the nearest 0.01 g

9.6 Spatula, wooden tongue depressors

9.7 Pasteur pipets, disposable, 2 mL

9.8 Boiling chips, PTFE, methylene chloride rinsed

9.9 Vials (14 mL), with PTFE-lined screw-cap lids, calibrated to 10 mL

9.10 Filter paper, qualitative: Fisher P8

9.11 N-EVAP concentrator

9.12 Syringes, micro: 100 uL, 500 μL, and 1000 μL

9.13 Aluminum foil

9.14 Filtration apparatus

9.14.1 Filter flask: 500 mL side-arm

9.14.2 Buchner funnel, 90 mm

9.14.3 Stopper: PTFE, holed

9.14.4 Glass-fiber filter paper for clay samples or samples with fine particulate

9.15 Filter funnel: 100 mm, ribbed

9.16 Sodium sulfate rinsing equipment:

9.16.1 Buchner funnel: 20 cm

9.16.2 Flask: 4 L vacuum

9.16.3 Drying pan: 13 x 9 inch, metal

9.16.4 Vacuum pump

9.16.5 Drying oven

9.16.6 Stopper, PTFE with hole for the Buchner funnel

9.16.7 Clamps, supporting

9.16.8 Filter paper, qualitative fast, 20 cm

9.16.9 Squirt bottle, PTFE, labeled with contents

9.17 Hot Plate: capable of variable temperature control to within ±5° C

9.18 Volumetric Flasks: 10 mL, 50 mL, 100 mL, 1 L

9.19 Amber vials, with PTFE-lined screw-cap lids, 40 mL

9.20 Analytical balances, capable of accurate measurement to the nearest 0.0001g

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Disrupters must be tuned at the beginning of each shift.

10.2 Ultrasonic probes must be cleaned before extracting each new sample and thoroughly cleaned at the end of each shift.

11.0 CHEMICALS AND REAGENTS

11.1 Methylene chloride/acetone, 1:1 (v/v): pesticides grade or better

11.2 Methylene chloride: pesticides grade or better

11.3 Sodium sulfate: ASC grade, anhydrous granular, rinsed:

11.3.1 This reagent must be rinsed before use. Assemble the sodium sulfate rinsing apparatus and clamp securely.

11.3.2 Insert filter paper in the Buchner funnel and add the entire contents of a 2.5 kg sodium sulfate container to the funnel.

11.3.3 Add 1 L of methylene chloride to the container, rinsing down the inside with methylene chloride from a PTFE squirt bottle.

11.3.4 Pour this methylene chloride over the sodium sulfate in the Buchner funnel, letting drain without applying vacuum. Add more methylene chloride if necessary to completely immerse the sodium sulfate.

11.3.5 After most of the solvent has drained, apply vacuum and rinse with an additional 100 mL from the PTFE squirt bottle. Maintain vacuum until solvent stops draining.

11.3.6 Transfer the rinsed sodium sulfate to a drying pan and heat in a drying oven at 120° C for at least one hour.

11.3.7 Remove from the oven with heat-resistant gloves and cool in a hood. Always place a caution sign by the pan while cooling.

11.3.7 After cooling, return to the original container using a glass funnel.

11.3.8 Label the container "Rinsed" with date and analyst's initials then place in reagent storage.

11.4 Hexane: Pesticides grade or better

11.5 Acetone: Pesticides grade or better

11.6 Each lot of methylene chloride, hexane and acetone must be tested before use to show freedom from interference. Submit test results to the quality assurance department for scanning into the certification of analysis file.

12.0 STANDARDS PREPARATION

12.1 Surrogates and spike solutions prepared from neat materials are made by the following rules:

12.1.1 Obtain an analytical balance that weighs to 0.0001 g. Be sure to record mass to the nearest 0.0001 g.

12.1.2 Place the balance in a fume hood and re-calibrate it.

12.1.3 Obtain glassware and materials required for surrogates or spike solutions being made.

12.1.4 Use the required solvent for dilutions.

12.1.5 Minimize headspace in storage vials.

12.2 Store surrogate and spiking solutions in 40 mL narrow-mouth amber vials, labeled with the following information:

12.2.1 Surrogate or spike name

12.2.2 Laboratory-assigned surrogate or spike ID

12.2.3 Date made

12.2.4 Analyst initials

12.2.5 Solvent

12.2.6 Concentration and units

12.2.7 Expiration date

12.3 If solid surrogate or spike compounds are weighed into a volumetric flask and the mass recorded, add enough solvent to dissolve then dilute to volume and invert several times to mix thoroughly.

Note: If solids do not dissolve, place in a small ultrasonic bath to aid in mixing. Do not leave for an extended time because the bath generates heat. After sonication, dilute to volume then transfer to a tightly capped pre-labeled vial and store at 4 ±2° C.

12.4 Weigh liquid surrogate or spike compounds into volumetric flasks containing approximately half-volume of solvent. Use a Pasteur pipet to add compound drop-wise, directly into the solvent. Be careful not to touch the inside of the flask or the solvent surface with the pipet tip. Record the mass and dilute to volume. Invert enough times to mix thoroughly then transfer to pre-labeled, tightly capped vials and store at 4 ±2° C.

12.5 Record surrogate/spike data in an extraction standards log (Attachment 20.1). Store surrogate/spiking solutions in the refrigerator at 4 ±2° C. A dilution of spiking solution must be analyzed by GC/MS and/or GC to check concentration prior to use by the extractions laboratory. Concentration must be within 80 – 120% and hardcopy submitted to the quality assurance department.

12.6 Neat compounds must be ACS grade or better. If the purity of a neat standard is below 95%, the concentration must be accounted for in all subsequent analytical calculations. Do not use any neat compound past its expiration date. If using the last of a compound, promptly re-order.

12.7 Shelf-life of surrogate and spiking solutions is six months for working solutions and one year for stock concentrations. Solutions will be disposed of sooner however if a manufacturer's expiration date occurs within that time for any neat material used. Once a chemical or solution has expired, remove from the laboratory for disposal. Monitor expiration dates of all chemicals and solutions. To avoid costly shipping, it is important to promptly order more chemical once consumed.

12.8 When making surrogate and spiking solutions, serial dilutions are often required to achieve the proper concentration. Serial dilutions are made from a higher concentration. The following example illustrates making a 1.0 mg/L working solution from a 10,000 mg/L stock concentration:

12.8.1 Using a micro-syringe, inject 1.0 mL of 10,000 mg/L solution into a 100 mL volumetric flask approximately half full of the appropriate solvent. Make sure the syringe needle tip is below the solvent surface. Fill to the mark with solvent then cap the flask and invert enough times to mix thoroughly. Concentration in the flask is 100 mg/L.

12.8.2 Inject 1.0 mL of the 100 mg/L solution into another volumetric flask half filled with solvent after rinsing the syringe thoroughly with clean solvent. Fill to the mark with solvent then cap the flask and invert enough times to mix thoroughly. Concentration in this second flask is 1.0 mg/L.

12.8.3 Concentration in the second flask is determined by the following calculation:

$$10000 \text{ mg/L x } \frac{1 \text{ mL stock standard}}{100 \text{ mL final volume}} = 100 \text{ mg/L}$$

$$100 \text{ mg/L x } \frac{1 \text{ mL stock standard}}{100 \text{ mL final volume}} = 1.0 \text{ mg/L}$$

12.9 When making a dilution, all data must be entered into the laboratory information management system (Element™). Enter each subsequent dilution in a dilution series separately. Use only pesticide grade (or better) solvents to dilute with.

12.10 Before using a laboratory-prepared solution, verify the calculations used by a second analyst. Once calculations have been verified, verify the solution concentration by actual analysis. If analytical recovery is not within laboratory established acceptance limits, a prepared solution cannot be used. Acceptance limits are 80 – 120% of expected value.

12.11 Dispose of vials containing 5 mL or less of a prepared solution at the end of the shift to minimize contamination and analyte concentration. When not in use, keep spike solution vials in the organic extractions refrigerator.

12.12 Surrogates are normally made from purchased solutions and diluted in acetone to make one 200 mL volume. Check by GC before using. If all surrogates in a spiking solution are within 80-120% of the expected value, the solution is approved for use. Remaining individual surrogate stock solutions are used to calibrate the GC.

Surrogate	Purchased Concentration (mg/L)	Stock Dilution (mL:mL)	Final Working Concentration (mg/L)
2,4,5,6-Tetrachloro-m-xylene	200	1:200	1
Decachlorobiphenyl	200	1:200	1

12.13 Pesticide analysis spike solution concentration:

12.13.1 Spikes are made from purchased solutions and diluted to 1.0 mg/L in hexane. Samples are spiked with 500 µL of the dilution to give extracts the same concentration as surrogates since final extract volume is 10.0 mL.

Pesticide Stock	Purchased Concentration	Dilution	Final Concentration
Purchased Mixture	1000 mg/L	80 µL/100 mL	0.8 mg/L

12.14 PCB spiking solutions are made from individually purchased Aroclors and diluted in hexane to make 100 mL working solutions, which are checked by GC before using. If the Aroclor in a working solution is within 80 - 120% of expected value when tested, the solution is approved for use. Stock solutions are also used to calibrate the GC. PCB spiking solutions are prepared by the GC analyst.

Aroclor	Purchased Concentration (mg/L)	Stock Dilution (mL:mL)	Final Concentration (mg/L)
1221	1000	1:100	10
1242	1000	1:100	10
1248	1000	1:100	10
1254	1000	1:100	10
1260	1000	1:100	10

13.0 ANALYTICAL PROCEDURE

13.1 The Fisher Model 550 ultrasonic disrupter must be tuned at the beginning of each shift, before samples are processed:

13.1.1 Turn the **OUTPUT CONTROL** knob counterclockwise to zero.

13.1.2 Press the **POWER SWITCH** to **ON** (up position). The switch will light up.

13.1.3 When the prompt appears, press **TUNE**. The display will read: [**TUNING - - - PROBE ACTIVE**].

13.1.4 Turn the **OUTPUT CONTROL** knob towards setting 10 (5 if using a microtip).

13.1.4.1 Note the position of the Bar Graph on the display screen. Do NOT exceed 70%.

13.1.4.2 Rotate the **OUTPUT CONTROL** knob clockwise or counterclockwise until a minimum (not maximum) reading is obtained. If a reading of less than 20% cannot be obtained, there is a problem with the disrupter and it must be repaired before using.

13.1.5 Press the **TUNE** key to display prompts for pulse or continuous operation and set the disrupter for pulse operation.

13.1.6 The disrupter is now tuned. Record all tuning information in the disrupter tuning logbook (Attachment 20.2).

13.2 Prior to sonication, homogenize all samples in accordance with the instructions outlined in TriMatrix SOP GR-09-128.

13.3 Label all glassware with sample number and other necessary information including MS, MSD, BS or BLK designations.

13.4 The low concentration extraction is as follows:

13.4.1 Perform the following steps rapidly to avoid loss of more volatile compounds

13.4.2 For sandy to dry samples, weigh 30 ±0.1 g of sample into 400 mL beakers and add 20 g to 25 g of anhydrous sodium sulfate. Mix well.

13.4.3 For wet to gummy samples, weigh 30 ±0.1 g of sample into 400 mL beakers and add 55 g to 60 g of anhydrous sodium sulfate. Mix well.

13.4.4 Samples should have a sandy texture after adding sodium sulfate. Add more sodium sulfate as necessary to achieve this sandy texture.

13.4.5 Measure 1.0 mL of surrogate into all samples and quality control extractions.

13.4.6 For matrix and blank spikes, add 0.5 mL of pesticides <u>or</u> 0.5 mL of PCB spiking solution.

13.4.7 Measure 100 mL of 1:1 methylene chloride/acetone into the beaker.

13.4.8 Sonicate by positioning the 3/4 inch sonication horn about 1/2 inch below the solvent surface without touching any sample solid or the beaker wall.

13.4.9 Sonicate for three minutes at full power (**OUTPUT CONTROL** knob set at 10 and **PERCENT-DUTY CYCLE** set at 50%). Do not use the microtip horn.

13.4.10 Next, assemble a K-D flask for each sample extracted to receive the extraction solvent.

13.4.11 Attach a 10 mL concentrator tube to the flask bottom.

13.4.12 Line 100 mm filter funnels with P8 filter paper then add approximately 1 inch of anhydrous sodium sulfate.

13.4.13 Decant the extraction solvent into the K-D flask through the funnel. Repeat with a different K-D setup for each sample extracted.

13.4.14 If a sample contains very fine particulate that plugs the filter paper, reweigh the sample and filter with a vacuum filter instead.

13.4.14.1 Assemble the vacuum filtration apparatus consisting of a 500 mL side-arm flask, 90 mm Buchner funnel and vacuum pump.

13.4.14.2 Filter using glass fiber filter paper under vacuum and transfer to a K-D flask for concentration.

13.4.15 Repeat the extraction twice more with additional 100 mL aliquots of 1:1 methylene chloride/acetone, decanting off the extraction solvent after each sonication. Repeat for each sample extracted until each sample has been extracted three times and the extract filtered.

13.4.16 If after the third sonication, there is no noticeable reduction in solvent color, up to two additional aliquots of methylene chloride/acetone may be used. Record the total number of aliquots used if more than 3.

13.4.17 After the final sonication, decant the solvent then transfer all solids into the filter funnel and quantitatively rinse with methylene chloride.

13.5 The high concentration extraction is as follows:

13.5.1 Perform the following steps rapidly to avoid loss of more volatile compounds

13.5.2 Weigh 2 ±0.1 g of sample into a clean, unused 20 mL vial.

13.5.3 Add 2 g anhydrous sodium sulfate to sample and mix well until free-flowing.

13.5.4 Add 1.0 mL surrogate to samples, blanks and spikes.

13.5.5 For pesticides analysis, add 0.5 mL of pesticide spike solution and 8.5 mL hexane to matrix spikes and laboratory fortified blanks. For PCBs analysis, add 0.5 mL of PCB spike solution and 8.5 mL hexane. The total volume added must be 10.0 mL.

13.5.6 Sonicate using the 1/8 inch tapered microtip horn for two minutes at **OUTPUT CONTROL** setting 5 in **PULSE MODE** and **PERCENT-DUTY CYCLE** set at 50%.

13.5.7 Filter the extract through glass wool and collect in a pre-calibrated 15 mL vial. Assume a final volume of 10 mL even though not all of the extract will be recovered. Do NOT bring to volume in the pre-calibrated vial.

13.5.8 Cap the vial tightly and store at 4 ±2° C until cleanup and/or analysis.

13.5.9 If cleanup is required, proceed to Section 13.9.

13.6 Extraction of wipe samples is as follows:

13.6.1 Measure 1.0 mL of surrogate solution into the jar a wipe sample arrives in. Do not remove any sample material from the jar. For extraction blanks and blank spikes, use a 400 mL beaker containing 30.0 g of sodium sulfate.

13.6.2 Add 0.5 mL of pesticide or 0.5 mL of PCB spike solution to matrix and blank spikes, depending upon the analysis required.

13.6.3 Add approximately 50 mL of hexane to each jar. The wipe sample must be totally covered by the solvent.

13.6.4 Position the 3/4 inch horn in the sample jar approximately 1/2 inch below the solvent surface, above the wipe.

13.6.5 Sonicate for three minutes at full power (**OUTPUT CONTROL** at ten and **PERCENT-DUTY** cycle at 50%).

13.6.6 Repeat Sections 13.4.10 – 13.4.19, except use hexane instead of 1:1 methylene chloride/acetone and rinse with hexane instead of methylene chloride.

13.6.7 Repeat for each sample needing extracted.

13.7 Concentrate extracts as follows:

13.7.1 Add one or two clean boiling chips to each concentrator tube and attach a three-ball Snyder column.

13.7.2 Pre-wet the Snyder column by adding approximately 1 mL of methylene chloride through the top.

13.7.3 Place the concentrator in a water bath (80-90° C) where the concentrator tube is partially immersed and the entire lower rounded surface of the K-D flask is bathed with vapor.

13.7.4 At the proper rate of distillation, the glass column balls will actively chatter but not flood. Until chattering begins, rattle the Snyder column periodically.

13.7.5 Concentrate to an apparent volume of 3 mL. The concentration takes ten to fifteen minutes for low concentration extracts and five to ten minutes for high concentration extracts).

Note: When solvent volume is reduced below 1 mL using the K-D flask, semi-volatile analytes can be lost. If extracts are concentrated to dryness or near-dryness, the entire extraction must be repeated.

13.8 Perform the hexane solvent exchange as follows:

13.8.1 Remove and cool. The volume in the concentrator tube after cooling should be approximately 8 mL. Add 50 mL of hexane, wet the Snyder column and evaporate as before.

13.8.2 Transfer the extract to a 14 mL vial calibrated to 10 mL, rinsing the concentrator tube with several aliquots of hexane, totaling approximately 2 mL. Use only enough to bring the final volume to 10.0 mL. Repeat for each sample extracted.

13.9 Extract Cleanup

13.9.1 If necessary, pesticide extracts may be cleaned up using Florisil® columns (1000 mg or 20 g) as described in TriMatrix SOP GR-09-111.

13.9.2 All PCB extracts are cleaned up with sulfuric acid (TriMatrix SOP GR-09-110), and copper (TriMatrix SOP GR-09-109). If necessary, they are further cleaned up with Florisil® (TriMatrix SOP GR-09-111) or Florisil®/silica Gel (TriMatrix SOP GR-09-120).

13.10 Store finished extracts at 4 ±2° C in the GC refrigerator.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Extraction analysts are responsible for sample documentation and data integrity. All documentation must be correctly filled in. It is important to document extractions by correctly filling in, turning in and filing all paperwork accurately. This is mandatory for quality control and to provide clients with defensible data.

14.2 Analysts extracting a batch of up to 20 samples must input all extraction data to Element™.

14.3 Benchsheets and logbooks must be filled in completely to ensure that results are reported correctly and data is associated with the right quality control batch (Attachment 20.3).

14.4 If an internal chain-of-custody report is required, it is important the form be filled in accurately and completely.

14.5 All extraction laboratory hardcopy must be archived appropriately.

14.6 All extraction logbooks must be filled in completely and correctly (Attachment 20.4). Corrections must be made with one line through the error, dated and initials then the correction to the side. Do not perform a write-over or obliterate the error. "Z" out blank areas in logbooks.

15.0 QUALITY ASSURANCE

15.1 Extract an extraction blank (BLK) and blank spike (BS) daily or once per shift to demonstrate that interferences and contamination are under control. Extract a replicate blank spike when insufficient sample volume is received to perform a matrix spike duplicate.

15.2 Blank spikes are prepared by adding 0.5 mL of spike solution and 1.0 mL surrogate spike solution to 30 g of sodium sulfate. Always add 1.0 mL of surrogate solution to blanks.

15.3 An extraction batch is limited to no more than 20 samples.

15.4 When sufficient sample volume is received, extract a matrix spike (MS) and matrix spike duplicate (MSD) with every batch of up to 20 samples.

15.4.1 Prepare a matrix spike by measuring 0.5 mL of PCB spike solution or 0.5 mL of pesticide spike solution to 30 g of sample. Extract a matrix spike in accordance with every stop in the extraction procedure.

15.4.2 Always add 1.0 mL of surrogate to matrix spikes before extraction.

15.5 Sample matrix interference or laboratory-induced contamination can affect analyte recovery. Investigation and re-extraction may be necessary if surrogate and/or spiked compounds fail to pass laboratory established acceptance limits.

15.6 Perform corrective action for out-of-control quality control samples in accordance with TriMatrix SOP GR-04-101.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Before preparation of actual samples, each analyst must demonstrate the ability to generate acceptable accuracy and precision by extracting an initial demonstration of capability (IDC) study.

16.2 Prepare a spiking solution to give a final extract concentration mid-range in the GC calibration. Prepare independently from the calibration. Prepare four 30 g aliquots of sodium sulfate then measure spiking solution to each. Extract as normal samples in accordance with all extraction steps in the procedure and all subsequent cleanup steps.

16.3 After extraction and cleanup, have the four extracts analyzed by TriMatrix SOP GR-03-128 or GR-03-120. Whichever is appropriate to the spiked analyte.

16.4 Input results to the IDC spreadsheet (located on the laboratory intranet library) to calculate average recovery and relative standard deviation.

16.4.1 Recovery must be within laboratory established control limits and relative standard deviation must be less than or equal to 20%. If all analytes and criteria are acceptable, the IDC study is complete. The analyst is authorized to extract samples by this procedure.

16.4.2 If one or more analytes fail, the analyst must proceed according through the following steps.

16.5 If any analyte fails, locate and correct the source of error then repeat the study successfully for the failed analyte.

16.6 Repeated failure will indicate a problem with the procedure and/or analyst technique. Locate the problem and correct the procedure and/or techniques used then repeat the IDC study successfully.

16.7 Samples may not be prepared by the analyst until an IDC study has been successfully completed.

16.8 Repeat the demonstration of capability study annually as a continuing demonstration of capability (CDC) by one of the follow approaches:

16.8.1 Repeat the initial demonstration of capability study.

16.8.2 Use the last four results from an MDL study run exclusively by the analyst. Input to the IDC spreadsheet and submit as the CDC.

Note: Results might be abnormal or unacceptable because of the low concentration. If so, repeat the study with a higher concentration of spikes.

16.8.3 Successfully complete a blind performance testing sample extracted during the course of routine sample extraction. Extraction of the sample must have been done by the analyst.

16.8.4 Use four consecutively extracted blank spikes extracted during the course of routine sample preparation. Extraction must have been done by the analyst.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in the laboratory to monitor their use.

17.2 Never dispose of laboratory chemicals without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount required.

18.3 Follow all instructions in SOP GR-15-102 for laboratory waste disposal requirements.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 3, February, 2007, Method 3550C, "Ultrasonic Extraction"

20.0 ATTACHMENTS

20.1 Standards Log Example

20.2 Disrupter Tuning Logbook Example

20.3 Preparation Batch Report Example

20.4 Extraction Logbook Example

Attachment 20.1
Standards Log Example

Analytical Standard Record
TriMatrix Laboratories, Inc.
7030451

Description	Chlorinated Pesticide Spike	Expires	Sep-12-07
Standard Type	Analyte Spike	Prepared	Mar-12-07
Solvent	MeOH Lot#044685	Prepared By	Andrea S. Colborn
Final Volume (mls)	100	Department	Semivolatiles GC
Vials	1	Last Edit	Apr-20-07 09:54 by JLW

Analyte	CAS Number	Concentration	Units
4,4'-DDD	72-54-8	0.8	ug/mL
4,4'-DDD [2C]	72-54-8	0.8	ug/mL
4,4'-DDE	72-55-9	0.8	ug/mL
4,4'-DDE [2C]	72-55-9	0.8	ug/mL
4,4'-DDT	50-29-3	0.8	ug/mL
4,4'-DDT [2C]	50-29-3	0.8	ug/mL
Aldrin	309-00-2	0.8	ug/mL
Aldrin [2C]	309-00-2	0.8	ug/mL
alpha-BHC	319-84-6	0.8	ug/mL
alpha-BHC [2C]	319-84-6	0.8	ug/mL
alpha-Chlordane	5103-71-9	0.8	ug/mL
alpha-Chlordane [2C]	5103-71-9	0.8	ug/mL
beta-BHC	319-85-7	0.8	ug/mL
beta-BHC [2C]	319-85-7	0.8	ug/mL
delta-BHC	319-86-8	0.8	ug/mL
delta-BHC [2C]	319-86-8	0.8	ug/mL
Dieldrin	60-57-1	0.8	ug/mL
Dieldrin [2C]	60-57-1	0.8	ug/mL
Endosulfan I	959-98-8	0.8	ug/mL
Endosulfan I [2C]	959-98-8	0.8	ug/mL
Endosulfan II	33213-65-9	0.8	ug/mL
Endosulfan II [2C]	33213-65-9	0.8	ug/mL
Endosulfan Sulfate	1031-07-8	0.8	ug/mL
Endosulfan Sulfate [2C]	1031-07-8	0.8	ug/mL
Endrin	72-20-8	0.8	ug/mL
Endrin [2C]	72-20-8	0.8	ug/mL
Endrin Aldehyde	7421-93-4	0.8	ug/mL
Endrin Aldehyde [2C]	7421-93-4	0.8	ug/mL
Endrin Ketone	53494-70-5	0.8	ug/mL
Endrin Ketone [2C]	53494-70-5	0.8	ug/mL

Reviewed By ____________ Date ____

Page 1 of 2

Analytical Standard Record
TriMatrix Laboratories, Inc.
7030451

gamma-BHC (Lindane)	58-89-9	0.8	ug/mL
gamma-BHC (Lindane) [2C]	58-89-9	0.8	ug/mL
gamma-Chlordane	5103-74-2	0.8	ug/mL
gamma-Chlordane [2C]	5103-74-2	0.8	ug/mL
Heptachlor	76-44-8	0.8	ug/mL
Heptachlor [2C]	76-44-8	0.8	ug/mL
Heptachlor Epoxide	1024-57-3	0.8	ug/mL
Heptachlor Epoxide [2C]	1024-57-3	0.8	ug/mL
Methoxychlor	72-43-5	0.8	ug/mL
Methoxychlor [2C]	72-43-5	0.8	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
A603424	(AMP) Organochlorine Pesticides	Feb-28-06	** Vendor **	Feb-29-08	Apr-20-07 09:54 by JLW	0.08

Reviewed By ____________ Date ____

Page 2 of 2

Attachment 20.2
Disrupter Tuning Logbook Example

Date	Analyst	Fisher Scientific 550 Inst. #1 Final Tuner Reading	Pass / Fail (<20%)	Fisher Scientific 550 Inst. #2 Final Tuner Reading	Pass / Fail (<20%)
7/16/02	DOM	4%	Pass / Fail	8%	Pass / Fail
7/17/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/18/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/19/02	JEO	4%	Pass / Fail	8%	Pass / Fail
7/22/02	[illegible]	[illegible]	Pass / Fail	[illegible]	Pass / Fail
7/23/02	DOM	5%	Pass / Fail	9%	Pass / Fail
7/24/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/25/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/26/02	JEO	5%	Pass / Fail	8%	Pass / Fail
7/27/02	DOM	4%	Pass / Fail	8%	Pass / Fail
7/29/02	DOM	5%	Pass / Fail	8%	Pass / Fail
7/30/02	JEO	5%	Pass / Fail	8%	Pass / Fail
8/1/02	JEO	5%	Pass / Fail	8%	Pass / Fail
8/5/02	DOM	7%	Pass / Fail	5%	Pass / Fail
8/8/02	JEO	5%	Pass / Fail	8%	Pass / Fail
8/9/02	JEO	5%	Pass / Fail	8%	Pass / Fail
8/12/02	JEO	4%	Pass / Fail	8%	Pass / Fail
8/13/02	JEO	4%	Pass / Fail	8%	Pass / Fail
8/14/02	JEO	4%	Pass / Fail	8%	Pass / Fail
8/15/02	DOM	5%	Pass / Fail	8%	Pass / Fail
8/16/02	JEO	4%	Pass / Fail	8%	Pass / Fail
8/20/02	DOM	6%	Pass / Fail	9%	Pass / Fail
8/21/02	DOM	5%	Pass / Fail	8%	Pass / Fail
8/22/02	DOM	5%	Pass / Fail	8%	Pass / Fail
8/23/02	DOM	[illegible]	Pass / Fail	7%	Pass / Fail
8/24/02	DOM	4%	Pass / Fail	8%	Pass / Fail
8/25/02	DOM	5%	Pass / Fail	7%	Pass / Fail
8/26/02	DOM	1%	Pass / Fail	9%	Pass / Fail
8/27/02	DOM	4%	Pass / Fail	8%	Pass / Fail
8/28/02	DOM	4%	Pass / Fail	8%	Pass / Fail

file: soniclog page: 16 of 50 revision. 1.0

Approved By: m 3-2-09
QA Officer

Approved By: BSH 3/2/09
Area Supervisor

Attachment 20.3
Preparation Batch Report Example

TriMatrix Laboratories, Inc.

PREPARATION BATCH 0709022 Page 1 of 1

Printed: 9/21/2007 5:34:48PM

Semivolatiles GC, Soil, 3550B Sonication Extraction

Surrogate #1 = 7070018 (Pre-Prep)

Batch Comments: (none)

Work Order	Analysis	Work Order	Analysis	Work Order	Analysis
0708116	8081A PESTs (master list)	0708257	8081A PESTs (master list)	0708330	8081A PESTs (master list)
0708338	8081A PESTs (master list)				

Lab Number	Contain	Prepared	By	Initial (g)	Final (mL)	uL Surrogate	Source ID	Spike ID	uL Spike	Client / QC Type	Extraction Comments
0709022-BLK1		Aug-08-07 07:57	BJH	30	10	1000				BLANK	
0709022-BS1		Aug-08-07 07:57	BJH	30	10	1000		7030451	500	LCS	
0709022-BSD1		Aug-08-07 07:57	BJH	30	10	1000		7030451	500	LCS DUP	
0708116-01	C	Aug-08-07 07:57	BJH	30	10	1000				[redacted]	[redacted]
0709022-BLK2		Aug-15-07 07:57	BJH	30	10	1000				BLANK	
0709022-BS2		Aug-15-07 07:57	BJH	30	10	1000		7030451	500	LCS	
0708257-01	C	Aug-15-07 07:57	BJH	30	10	1000				[redacted]	[redacted]
0709022-BLK3		Aug-21-07 07:57	BJH	30	10	1000				BLANK	
0709022-BS3		Aug-21-07 07:57	BJH	30	10	1000		7030451	500	LCS	
0708330-04	A	Aug-21-07 07:57	BJH	30	10	1000				[redacted]	[redacted]
0708338-01	C	Aug-21-07 07:57	BJH	30	10	1000				[redacted]	[redacted]
0708338-02	C	Aug-21-07 07:57	BJH	30	10	1000				[redacted]	[redacted]

Comments:	Analyst Initials:

bch_TriMatrix.rpt

Approved By: M 3-2-09
QA Officer

Approved By: BJH 3/2/09
Area Supervisor

Attachment 20.4
Extractions Logbook Example

TriMatrix Laboratories, Inc.

Organic Extraction Lab
Soil Extraction Logbook

Date: 3/11/04

Client	Project/ Submittal	Sample Number Range	LIMS Batch ID	Test or Parameter	Surrogate Spike Number and Volume		Matrix Spike Number and Volume	
EQ	36357-12	355751-756	212470	8082 PCB 3550	PL4.1-10	1.0mL	PL4.3-2	500uL
SAIC	36278-43	356038-046	213415	8330 Exp	PL3.12-2	500 uL	PL4.2-13	1.0 mL
MDL				Full List Plus	PL4.2-7	100 uL	PL3.12-12 PL3.11-12	5 uL 1 uL
				PAK	3/11/04			

LIMS Sample ID	Bath Temp. °C	Initial Volume (g)	Final Volume (mL)	Analyst Initials	LIMS Sample ID	Bath Temp. °C	Initial Volume (g)	Final Volume (mL)	Analyst Initials
MPB111	90	30.0	10.0	MBS	MDL 1	80	30.0	1.0	MBS
355751		30.0			MDL 2		30.0		
355752		30.0			MDL 3		30.0		
355753		30.0			MDL 4		30.0		
355754		30.0			MDL 5		30.0		
355755		30.0			MDL 6		30.0		
355756		30.0			MDL 7		30.0		
MPB111	N/A	2.0	10.0	DJM					
356038		2.0							
356039		2.0					PAK		
356040		2.0							
356041		2.0					3/11/04		
356042		2.0							
356043		2.0							
356044		2.0							
356045		2.0							
356046		2.0							
356042MS		2.0							
356042MSD		2.0							
LFB111		2.0							
			PAK 3/11/04						

Reagent Information

CH$_2$Cl$_2$ Lot #: 030986
Hexane Lot #: 030702
Acetonitrile Lot #: Y30819
1:1 CH$_2$Cl$_2$/Acetone Reagent #: PLR1.48-7
Na$_2$SO$_4$ Reagent #: PLR1.51-2
Other #: N/A

file: Prep Lab Soil Logbook page: 4 of 20 revision: 0.0

Approved By: [signature] 3-2-09
QA Officer

Approved By: BJH 3/2/09
Area Supervisor

TriMatrix
Laboratories, Inc.

STANDARD OPERATING PROCEDURE

Sulfur Cleanup

SW-846 Method 3660B

APPROVALS:

Area Supervisor: Brian J. Hall Date: 3/2/09

QA Officer: Tom C. Boocher Date: 2-27-09

Operations Manager: Jeff P. Glaser Date: 3/3/09

Procedure Number: GR-09-109
Revision Number: 3.3

Date Initiated: 3/30/94
Effective Date: 3/27/09

Date Revised: 2/27/09
Pages Revised: All

By: Daniel J. Mierendorf
Total Number of Pages: 8

If signed below, the last annual review required no procedural revision.

Date Reviewed	Reviewed by	Review Expires

1.0 SCOPE AND APPLICATION

1.1 Elemental sulfur is encountered in many sediment samples, marine algae and industrial wastes.

1.2 Sulfur solubility in various solvents is similar to that of organochlorine pesticides and polychlorinated biphenyls (PCB) and is an interferent in gas chromatographic detection.

1.3 All PCB extracts must undergo sulfur cleanup prior to analysis.

1.4 Sulfur cleanup in organochlorine pesticide extracts has limited applications and must only be used when analyzing for specific analytes shown not to be affected by copper (Refer to Attachment 20.2). Before cleaning up a pesticide analyte not on the list, a demonstration of capability study must be performed to verify acceptable recovery.

2.0 PRINCIPLE METHOD REFERENCES

2.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 2, December, 1996, Method 3660B, "Sulfur Cleanup"

3.0 SUMMARY OF PROCEDURE

3.1 Sample extracts are mixed with reactive copper granules and vortexed to remove sulfur.

3.2 As the sulfur is removed, the copper turns black.

4.0 PARAMETER OR COMPOUND LIST

4.1 Refer to TriMatrix SOP GR-03-120 and GR-03-128 for parameter lists.

5.0 REFERENCED SOPs

5.1 TriMatrix SOP GR-03-128, *Semi-Volatile Laboratory Gas Chromatography Analysis of Polychlorinated Biphenyls (PCBs)*, latest revision

5.2 TriMatrix SOP GR-03-120, *Semi-Volatile Laboratory Gas Chromatography Analysis of Pesticides*, latest revision

5.3 TriMatrix SOP GR-15-102, *Laboratory Waste Disposal*, latest revision

5.4 TriMatrix SOP GR-16-100, *Equipment Cleaning and Preparation for the Organics Extraction Laboratory*, latest revision

6.0 INTERFERENCES AND CORRECTIVE PROCEDURES

6.1 This technique requires that copper granules be reactive as indicated by a bright and shiny appearance to maximize sulfur removal. Refer to Section 10.0 for instructions on oxide removal.

6.2 Interference can be caused by contaminants in solvents, reagents, glassware or sample processing equipment leading to discrete artifacts and/or elevated baselines. Extraction blanks must be run to monitor such laboratory interferences.

6.3 Always use pesticide grade solvents and ACS grade chemicals or better.

6.4 Clean glassware in accordance with TriMatrix SOP GR-16-100.

6.4 Phthalate esters cause interference with pesticide and PCB analyses. Avoid using flexible plastics in contact with solvents to minimize phthalate ester contamination.

6.5 Sample matrix interference can affect analyte recovery. Repeating sample cleanup may be necessary if surrogates and/or other spiked compounds fail laboratory acceptance limits.

7.0 SAFETY PRECAUTIONS

7.1 A laboratory coat, disposable gloves and approved safety glasses must be worn when working in the organic extractions laboratory.

7.2 Follow all instructions as outlined in the TriMatrix Laboratory Safety Manual and Chemical Hygiene Plan.

7.3 Refer to TriMatrix SOP GR-15-102 for laboratory waste disposal.

7.4 The toxicity of chemicals used in this procedure has not been precisely defined.

7.4.1 All chemicals must be treated as a potential health hazard.

7.4.2 Exposure must be reduced to the lowest possible level by adherence to established safety practices.

7.4.3 Refer to the material safety data sheet if there is a question about chemical handling. Material safety data sheets are located on the laboratory intranet library.

7.5 Bring all safety issues to the attention of the area supervisor and/or the health and safety officer.

8.0 SAMPLE SIZE, COLLECTION, PRESERVATION AND HANDLING PROCEDURES

8.1 There is no sample handling directly associated with this procedure.

9.0 INSTRUMENTATION, APPARATUS AND MATERIALS

9.1 Vortex Genie or equivalent

9.2 Pasteur pipettes, glass, 1 mL, disposable

9.3 Autosampler vials, 1.5 mL

9.4 Spatulas

10.0 ROUTINE PREVENTIVE MAINTENANCE

10.1 Copper powder must be reactive and have a bright and shiny appearance. Copper oxidation reduces the effectiveness of sulfur cleanup and needs to be removed as follows:

10.1.1 Rinse a small portion of copper powder in dilute nitric acid to remove the dull surface. Swirl the mixture to achieve good surface contact of the powder with acid.

10.1.2 When the copper appears bright and shiny, decant the acid and rinse thoroughly with organic-free laboratory reagent water. Rinse again with acetone to remove most of the water.

10.1.3 Carefully dry with clean nitrogen gas.

10.1.4 Repeat as necessary.

11.0 CHEMICALS AND REAGENTS

11.1 Copper powder, granular, 10-40 mesh

11.2 Nitric acid, diluted 1:10 (v/v) with laboratory reagent water

11.3 Laboratory reagent water, organic-free

11.4 Acetone, pesticide grade or better

11.5 Nitrogen gas, oil-free

12.0 STANDARDS PREPARATION

12.1 There is no standards preparation directly associated with this procedure.

13.0 ANALYTICAL PROCEDURE

13.1 Add 0.5 to 1.0 g of bright and shiny copper granules to 1.0 mL of sample extract.

13.2 Vortex for approximately one minute.

13.3 If the copper turns black, use a disposable Pasteur pipette to transfer the extract to a new vial then add fresh copper powder and repeat. A color change to black indicates sulfur is present. Repeat the copper addition until the copper remains bright and shiny.

13.4 Do NOT add more solvent as the volume remaining still represents the 1.0 mL final volume and is ready for further cleanup and/or analysis.

14.0 DATA REPORTING AND DELIVERABLES

14.1 Analysts are responsible for documenting sample cleanup by correctly and completely filling in the PCB cleanup logbook. This is mandatory for quality control and to provide clients with defensible data.

14.2 The cleanup logbook must be filled in completely and correctly. Corrections must be made with a line-out and not a write-over or scribble-out. Blank lines in the logbook need to be Z'd out and initialed.

15.0 QUALITY ASSURANCE

15.1 Be sure the copper used is clean and reactive. If uncertain, rinse with nitric acid as outlined.

15.2 Centrifuge extracts containing visible sulfur crystals then transfer to a new vial before adding copper powder. Samples with interference on chromatograms after sulfur cleanup may need the sulfur cleanup repeated.

15.3 Surrogate recovery must be within laboratory established acceptance limits after sulfur cleanup.

15.4 All extracted quality control associated with samples using this procedure must go through the sulfur cleanup.

16.0 DEMONSTRATIONS OF CAPABILITY/METHOD VALIDATION

16.1 Demonstration of capability studies for PCB extraction procedures must include sulfur cleanup.

17.0 POLLUTION PREVENTION

17.1 Maintain an inventory of all chemicals used in this procedure to monitor their use.

17.2 Never dispose of a laboratory chemical without first referencing appropriate written instructions of disposal for that particular material.

17.3 Conserve the use of chemicals where applicable.

17.4 Comply with all environmental laws associated with chemicals in the laboratory.

18.0 WASTE MANAGEMENT

18.1 Consult the appropriate Material Safety Data Sheet (MSDS) when disposing of chemicals.

18.2 To minimize the environmental impact and costs associated with chemical disposal, order and use only the minimum amount of material required.

18.3 Follow all instructions in SOP GR-15-102 for laboratory waste disposal.

19.0 REFERENCES

19.1 *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition,* Final Update IV, Revision 2, December, 1996, Method 3660B, "Sulfur Cleanup"

20.0 ATTACHMENTS

20.1 PCB Cleanup Logbook

20.2 Effects of Copper Granules on PCB and Pesticide Recoveries

Attachments 20.1
PCB Cleanup Logbook

TriMatrix Laboratories, Inc.

PCB Cleanup Logbook

Date	Analyst	Client	Sample Numbers	H_2SO_4	Copper	Combined Cleanup Using 1g Florisil/ 1g Silica Gel/Sodium Sulfate Plug: Florisil	Silica Gel	Sodium Sulfate	Other (Largo Florisil, Cartridge, etc.) Include Reagent Numbers and g Used
6/20/07	BJH	[redacted]	0706314-01, BLK, BS	PLC1.57-4	7050086				
		[redacted]	0706348-19						
		[redacted]	0706259-02						
		[redacted]	0706261-01-02						
		[redacted]	0706242-01						
		[redacted]	0706243-01-02						
		[redacted]	0706365-01-03, BLK, BS						
6/26/07	BJH	[redacted]	0706494-01-02, BLK, BS						
~~6/27/07~~	KMB	[redacted]	BLK, BS 0706504-01						
6/27/07	KMB	[redacted]	0706535-01	PLR1.574	7050086				
6/28/07	ASC	[redacted]	BLK, BS, 0706542-01						
		[redacted]	BLK, BS, 0706504-01 0706504-02			PLR1.31-10	7020101		
6/28/07	BJH	[redacted]	0706579-01, BLK, BS						
7/2/07 ~~6/29/07~~	BJH	[redacted]	0706549-01, BLK, BS						
		[redacted]	0706410-01RE, 14RE, 15RE, BLK, BS						
		[redacted]	0706518-01						

file: pcblogbook1 page: 4 of 25 revision: 1.2

Approved By: [signature] 3-2-09
QA Officer

Approved By: BJH 3/2/09
Area Supervisor

Attachment 20.2
Effects of Copper on PCB and Pesticide Recoveries

EFFECT OF COPPER ON PESTICIDES

Pesticide	Percent Recovery[a] Using Copper
Aroclor 1254	104.26
Lindane	94.83
Heptachlor	5.39
Aldrin	93.29
Heptachlor epoxide	96.55
DDE	102.91
DDT	85.10
BHC	98.08
Dieldrin	94.90
Endrin	89.26
Chlorobenzilate	0.00
Malathion	0.00
Diazinon	0.00
Parathion	0.00
Ethion	0.00
Trithion	0.00

a Percent recoveries cited are averages based on duplicate analyses for all compounds other than for Aldrin and BHC. For Aldrin, four and three determinations were averaged to obtain the result for copper. Recovery of BHC using copper is based on one analysis.

APPENDIX C

EXAMPLE CHAIN-OF-CUSTODY FORM

TriMatrix Laboratories, Inc.

5560 Corporate Exchange Court SE Grand Rapids, MI 49512
Phone (616) 975-4500 Fax (616) 942-7463
www.trimatrixlabs.com

Chain of Custody Record

COC No. 130613

Page 1 of 1

For Lab Use Only

Cart:

VOA Rack/Tray:

Receipt Log No. 35.13

Project Chemist:

Laboratory Project No. 0910334

Client Name	Weston Solutions, Inc.
Address	20 N. Wacker Dr., Suite 1210, Chicago, IL 60606
Phone	312-424-3339
Fax	312-424-3330
Project Name	Allied Paper - No. 2 Dam
Client Project No./P.O. No.	20405.012.001.0778.00
Invoice No.	☐ Client ☐ Other (comments)
Contact/Report To	Lisa Graczyk

Analyses Requested: PCBs

← PRESERVATIVES
- A NONE pH~7
- B HNO_3 pH<2
- C H_2SO_4 pH<2
- D 1+1 HCl pH<2
- E NaOH pH>12
- F ZnAc/NaOH pH>9
- G MeOH
- H Other (note below)

Container Type (corresponds to Container Packing List)

Test Group	Matrix Code	Laboratory Sample Number		Sample ID	Cooler ID	Sample Date	Sample Time	COMP	GRAB	Matrix	Number of Containers Submitted (PCBs)	Total	Sample Comments
		-01	1	PD2-102009-05-SD/TS20322	—	10/20/09	1511	X		Sed.	✓	1	
			2										
			3										
			4										
			5										
			6										
			7										
			8										
			9										
			10										

EXAMPLE

Sampled By (print): Michael Browning

Sampler's Signature: Michael J. Browning

Company:

How Shipped? Hand Carrier ____________

Tracking No.

Comments: 24-hour TAT

1. Relinquished By	Date	Time	2. Relinquished By	Date	Time	3. Relinquished By	Date	Time
Michael J. Browning	10/20/09	1638						
1. Received By	**Date**	**Time**	**2. Received By**	**Date**	**Time**	**3. Received For Lab By**	**Date**	**Time**
						[signature]	10/20/09	1642

TriMatrix Laboratories, Inc.

EXAMPLE
SAMPLE RECEIVING / LOG-IN CHECKLIST

Client: Weston Solutions		New / Add To	Project-Submittal No. 0910334
Receipt Record Page/Line No. 35-13		Project Chemist: MH	Sample Nos. 01

Coolers Received

Recorded by (initials/date)		Qty Received	Thermometer Used	
LR 10/20/09	☒ Cooler ☐ Box ☐ Other___	1	☒ IR Gun (#202) ☐ Digital Thermometer (#54) ☐ Other (#______)	☐ See Additional Cooler Information Form

	Cooler 1	Cooler 2	Cooler 3	Cooler 4
Cooler No.	—			
Time	1645			
Custody Seals:	☒ None ☐ Present / Intact ☐ Present / Not Intact	☐ None ☐ Present / Intact ☐ Present / Not Intact	☐ None ☐ Present / Intact ☐ Present / Not Intact	☐ None ☐ Present / Intact ☐ Present / Not Intact
Coolant Location:	Dispersed / Top / Middle / Bottom	Dispersed / Top / Middle / Bottom	Dispersed / Top / Middle / Bottom	Dispersed / Top / Middle / Bottom
Coolant/Temperature Taken Via:	☐ Loose Ice / Avg 2-3 containers ☒ Bagged Ice / Avg 2-3 containers ☐ Blue Ice / Avg 2-3 containers ☒ None / Avg 2-3 containers (LR 10/20)	☐ Loose Ice / Avg 2-3 containers ☐ Bagged Ice / Avg 2-3 containers ☐ Blue Ice / Avg 2-3 containers ☐ None / Avg 2-3 containers	☐ Loose Ice / Avg 2-3 containers ☐ Bagged Ice / Avg 2-3 containers ☐ Blue Ice / Avg 2-3 containers ☐ None / Avg 2-3 containers	☐ Loose Ice / Avg 2-3 containers ☐ Bagged Ice / Avg 2-3 containers ☐ Blue Ice / Avg 2-3 containers ☐ None / Avg 2-3 containers
Alternate Temperature Taken Via:	☐ Temperature Blank (TB) ☐ 1 Container	☐ Temperature Blank (TB) ☐ 1 Container	☐ Temperature Blank (TB) ☐ 1 Container	☐ Temperature Blank (TB) ☐ 1 Container

	Recorded °C	Correction Factor °C	Actual °C	Recorded °C	Correction Factor °C	Actual °C	Recorded °C	Correction Factor °C	Actual °C	Recorded °C	Correction Factor °C	Actual °C
Temp Blank:												
TB location: Representative / Not Representative												
1	15.4	—	15.4									
2												
3												
Average °C			15.4									

☐ Cooler ID on COC? ☐ VOC Trip Blank received? (each cooler)

If any shaded areas checked, complete Sample Receiving Non-Conformance Form

Paperwork Received

☐ No COC Received

N/A	Yes	No	
	☒	☐	Chain of Custody record(s)?
	☐		If No, COC Initiated By ______
	☒		Rec'd for Lab Signed/Date/Time?
☒	☐		Shipping document?
☒	☐		Other ______

COC ID Nos.

☒ TriMatrix 130613

☐ Other (Name or ID#)______

Check COC for Accuracy

☐ No analysis requested

Yes	No	
☒	☐	Sample ID matches COC?
☒	☐	Sample Date and Time matches COC?
☒		Container type completed on COC?
☒	☐	All container types indicated are received?

Sample Condition Summary

☐ Non-TriMatrix containers, see Notes

N/A	Yes	No	
	☐	☒	Broken containers/lids?
	☐	☒	Missing or incomplete labels?
	☐	☒	Illegible information on labels?
	☐	☒	Low volume received?
	☐	☒	Inappropriate containers received?
☒	☐	☐	VOC vials / TOX containers have headspace?
☒	☐	☐	Extra sample locations / containers not listed on COC?

Check Sample Preservation

N/A	Yes	No	
	☐	☒	Average sample temperature ≤6° C?
☒	☐		Completed Sample Preservation Verification Form?
☒	☐	☐	Samples preserved correctly?
☒	☐		If "No", added orange tag?
☒	☐		Received pre-preserved VOC soils?

☐ MeOH ☐ Na_2SO_4

Check for Short Hold-Time Prep/Analyses

- ☐ Bacteriological
- ☐ Air Bags
- ☐ EnCores / Methanol Pre-Preserved
- ☐ Formaldehyde/Aldehyde
- ☐ Green-tagged containers
- ☐ Yellow/White-tagged 1L ambers (SV Prep-Lab)

AFTER HOURS ONLY:
COPIES OF COC TO LAB AREA(S)
☐ NONE RECEIVED
☒ RECEIVED, COCs TO LAB(S)

Notes

☐ Trip Blank received ☐ Trip Blank not listed on COC

☐ No COC received, Proj. Chemist reviewed (Init/Date) ______

☐ No analysis requested, Proj. Chemist completed (Init/Date) ______

Cooler Received (Date/Time)	Paperwork Delivered (Date/Time)	≤1 Hour Goal Met?
10/20/09 16:42	10/20/09 1646	Yes / No

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

SRT-5J

MEMORANDUM

DATE: June 10, 2010

SUBJECT: Approval of the Removal Quality Assurance Project Plan (QAPP) for Allied Paper Plainwell #2 Dam PRP PCB Removal Site, Plainwell, Michigan

FROM: Ida Levin, Quality Assurance Team Leader
Remedial Response Section 2

TO: Sam Borris, On-Scene Coordinator (OSC)

I am providing approval of the First Revision of the Removal Quality Assurance Project Plan (QAPP) for Allied Paper Plainwell #2 Dam PRP PCB Removal Site, Plainwell, Michigan. The document was received on May 19, 2010 (SF Log-in No.3921).
The contractor addressed all EPA comments.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

SRT-5J

MEMORANDUM

DATE: October 26, 2009

SUBJECT: Review of the Removal Quality Assurance Project Plan (QAPP) for Allied Paper Plainwell #2 Dam PRP PCB Removal Site, Plainwell, Michigan

FROM: Ida Levin, Quality Assurance Team Leader
Field Services Section (FSS)

TO: Sam Borris, On-Scene Coordinator (OSC)

I have reviewed the Removal Quality Assurance Project Plan (QAPP) for Allied Paper Plainwell #2 Dam PRP PCB Removal Site, Plainwell, Michigan prepared by Weston Solution. The document was received by FSS on October 19, 2009 (SF Log-in No.3833).

The following needs to be addressed in the submitted QAPP:

1. The signature page should include name of the OSC.
2. Worksheet #5. The name of the OSC should be changed.
3. Worksheet #9. The name of the OSC should be changed.
4. Worksheet #16. The dates for the split sampling activities should be corrected.
5. The Laboratories SOPs and Chain of Custody procedures should be included in the QAPP.